

THERMAL ANALYSIS OF SOME SCCL COALS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE
DEGREE OF

BACHELOR OF TECHNOLOGY IN MINING ENGINEERING

BY

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**DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA - 769008
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2010**



National Institute of Technology Rourkela

CERTIFICATE

This is to certify that the thesis entitled “**THERMAL ANALYSIS OF SOME SCCL COAL**” submitted by **Sri M.Bharath Kumar** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ABSTRACT

The auto oxidation of coal ultimately leads to spontaneous combustion which is the major root cause for the disastrous of coal mine. It has been a major problem in the leading producing coal countries like Australia, India and China. Therefore the assessment for this combustion is very much necessary. It depends upon different characteristics and properties of coal. Once if the combustion of coal has been occurred, it is very difficult to control which also disturbs the environment of the surroundings of the mine. The spontaneous heating susceptibility of different coals varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic aspects of mining etc

The B.Tech.dissertation deals with the thermal analysis of various parameters of coal with the spontaneous heating tendency of coal. Nine insitu coal samples for the project were collected from Singareni Collieries Company Ltd. (SCCL), both from opencast as well as underground workings. The project deals with determination of spontaneous heating susceptibility of coal samples by experimental techniques. The intrinsic properties as well as susceptibility indices of the coal samples were determined by following experimental techniques:

- Proximate analysis
- Calorific value
- Flammability temperature
- Wet oxidation potential
- Differential thermal analysis (DTA-TG)

It was interpreted from the analysis that DTA – TG is the best method to assess the spontaneous heating tendency of coal in comparison to Flammability temperature and Wet oxidation potential. It was also observed that the Transition temperature obtained from the DTA – TG plot cannot be taken as a sole parameter to assess the spontaneous heating of coal, rather Stage IIB

and Stage II slopes give a better idea. It was also found that Flammability temperature method does not provide an accurate measure of spontaneous heating tendency of coal.

It was found that Wet oxidation potential gave a fair enough approximation for spontaneous heating tendency for high moisture coals. However it can be taken as a parameter for spontaneous heating susceptibility as it requires less time for conduction than DTA –TG.

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CHAPTER – 1

INTRODUCTION

1.1 Background

Coal is the source of about 27% of the world's primary energy consumption and it accounts for about 34% of electricity generated in the world, so much attention has been focused in recent years on coal as an alternative source of energy (Nimaje et.al., 2010). Coal is the dominant energy source in India and meets 55% of country's primary commercial energy supply. Mine fires in Indian coalfields is generally caused by spontaneous combustion of coal despite various preventive technologies being adopted. The spontaneous heating of coal varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic aspects of mining (Tripathy, 2001).

In order to find out the susceptibility of coal to spontaneous heating different methods have been adopted by various researchers of the world. A number of experiments have been done for assessing the spontaneous heating susceptibility of coal viz., Crossing point temperature method (Didari et.al., 2000), Wet oxidation potential method (Tarafadar et.al., 1989), Differential thermal analysis (Nimaje et.al., 2010), Flammability temperature (Nimaje et.al., 2010). A number of approaches have been developed over the years to assess the proneness of coal to spontaneous heating. This propensity to self heating of coal also decide the incubation period of coal seam, which decide the size of the panel to be formed, which is a most important safety measure in mine planning. It is therefore imperative that the planners of a mine determine in advance the spontaneous heating susceptibility of the seam/seams to be mined so that either the coal has been extracted before the incubation period, or advance precautionary measures are planned to tackle this menace.

The methods used to assess the tendencies of coals to spontaneous heating in the present study are Proximate analysis, Calorific value, Flammability temperature, Wet oxidation potential, Differential thermal analysis (DTA - TG).

1.2 Objectives of the project

The objectives of the project is to carry out thermal analysis of all the parameters of coal in order to find out the mostly affected collected coal sample susceptible to spontaneous heating. The project was divided into the following steps to achieve the abovesaid objective –

- **Literature review** – collection of all the past works done by various academicians/researchers/scientists both national and international.
- **Sample collection and preparation** – Nine samples were collected from SCCL, for the purpose of analysis and the samples were collected and prepared as per the Indian Standards.
- **Experimentation** – the experimentation part divided into two stages:
 - Determination of intrinsic properties of coal – proximate analysis, calorific value
 - Determination of susceptibility indices of coal – wet oxidation potential, differential thermal analysis, flammability temperature.
- **Analysis** – Different graphs have been plotted between the intrinsic properties and susceptibility indices and the interpretation has been carried out on the basis of graphs.

CHAPTER – 2

LITERATURE REVIEW

2.1 Coal mine fires

Mine fires are associated mostly with coal mines, though fires in pyrite mines and occasional timber fires in certain metal mines are not unknown. Mine fires are common occurrences in coal mines but are rare in metal mines. Mine fires can be caused either by spontaneous heating, explosion of gases, electrical failures and blasting. In coal mines the major cause of mine fire is spontaneous heating of coal. An analysis of the causes of coal mine fires reveal that they may start either from open fires over the external mining agencies or originate due to very nature of coal (Pal et.al., 1998). The propensity of coal liberating heat when come in contact with oxygen of air and its poor thermal conductivity favoring heat accumulation, may give rise to latter kind of heating. The former type of fire from external agencies is known as Exogenous Fires and the latter type i.e. due to self-heating characteristics of coal is called Endogenous Fires or Spontaneous Combustion.

Endogenous fires:

1. Pyrite fires: The iron ore of pyrite having chemical formula of FeS_2 is a polysulphide of iron. Pure pyrite contains 46.37% Fe and 53.33% S. as with coal pyrite also reacts with oxygen of air at room temperature liberating heat which under favorable conditions of heat accumulation gives rise to spontaneous fires. Susceptibility to spontaneous heating of pyrites is much less than that of coal but it increases if carbonaceous materials are present in pyrites. There are instances when pyrites with 5 - 6 % C and 10 – 12 % S have caught fire due to spontaneous heating (Amjhore Field, India).

2. Endogenous heating timber: Decayed timber may under extremely favorable conditions give rise to spontaneous heating believed to be mainly from bacterial origin.

Exogenous fires:

Electricity is one of the important causes of mine fires. It may originate from short circuiting, over heating of machines, electric bulbs, candles, flames from fires or explosion while blasting and ignition of inflammable materials like timber, oil or wastes. At times crushing of sulphide ores or fires from surface may also be the origin.

2.2 History of coal mine fires

Self heating of coal leading to spontaneous combustion is the most significant cause of fires in coal mines across the world (Ramlu et.al., 1985). Self heating of coal can occur in underground mines, opencast mines, coal stockpiles, transportation and during the disposal of wastes from coal using industries in heap wastes (Bowes, 1984 and Carras et.al., 1994).

World scenario

Up to 10 coal fires per year in the Ruhr area of Germany are caused by spontaneous heating (Pilarczyk et.al., 1995). In china underground coal fires are widespread within a region stretching 5000 Km east – west and 750 Km north – south. It is assumed that fires in northern China consume an estimated amount of 100 – 200 MT of underground coal which is about 2 – 3 % of world CO₂ production (Huang et.al., 2001).surveys in the West Riding of Yorkshire (England) showed that 45 of the county's 153 collieries were on fire in 1931 (Sheail, 2005). A more recent example is the spontaneous combustion of spoil heaps at Middleburg colliery in Witbank coalfield in South Africa (Bell et.al., 2001).

China

In China, the world's largest coal producer with an annual output around 2.5 billion tons, coal fires are a serious problem. It has been estimated that some 10-20 million tons of coal uselessly burn annually, and that the same amount again is made inaccessible to mining. They are concentrated in the provinces of Xinjiang, Inner Mongolia and Ningxia. Beside losses from burned and inaccessible coal, these fires contribute to air pollution and considerably increased levels green house gas emissions and have thereby become a problem which has gained international attention^[32].

Germany

In Planitz, now a part of the city of Zwickau, a coal seam that had been burning since 1476 could only be quenched in 1860. In Dudweiler (Saarland) a coal seam fire ignited around 1668 and is still burning today. Also well-known is the so-called Stinksteinwand (stinking stone wall) in Schwalbenthal on the eastern slope of the Hoher Meibner, where several seams caught fire centuries ago after lignite coal mining ceased; combustion gas continues to reach the surface today^[32].

Indonesia

Coal and peat fires in Indonesia are often ignited by forest fires near outcrop deposits at the surface. No accurate count of coal seam fires has been completed in Indonesia. Only a minuscule fraction of the country has been surveyed for coal fires. The best data available come from a

study based on systematic, on-the-ground observation. In 1998, a total of 125 coal fires were located and mapped within a 2-kilometer strip either side of a 100-kilometer stretch of road north of Balikpapan to Samarinda in East Kalimantan, using hand-held Global Positioning System (GPS) equipment. Extrapolating this data to areas on Kalimantan and Sumatera underlain by known coal deposits, it was estimated that more than 250,000 coal seam fires may have been burning in Indonesia in 1998^[32].

United States

Many coalfields in the USA are subject to spontaneous ignition. The Federal Office of Surface Mining (OSM) maintains a database (AMLIS), which in 1999 listed 150 fire zones. In Pennsylvania, 45 fire zones are known, the most famous being the fire in the Centralia mine in the hard coal region of Columbia County. In Colorado coal fires have arisen as a consequence of fluctuations in the groundwater level, which can increase the temperature of the coal up to 30 °C, enough to cause it to spontaneously ignite. The Powder River Basin in Wyoming and Montana contains some 800 billion tons of brown coal, and already the Lewis and Clark Expedition (1804 to 1806) reported fires there^[32].

India



Plate 2.1 Coal reserves of India

History of coal mines fires can be traced back to the year 1865, when the first fire was reported in Raniganj Coalfields. Over 140 years fires have been reported till the year 1967 from both Jharia and Raniganj coal fields and superior quality non-cocking coal in Raniganj coal fields. Fires occur whenever and wherever combustible material is present in mine working. They endanger not only the valuable lives of men in mine but also cause considerable economic losses to the organization affected by them. These fires not only continue to spread to adjoining areas, adding to the losses but also prevent economic exploitation of the seam in the vicinity. Again the open fire in these fields causes environmental pollution by emission of huge quantities of steam, smoke and noxious gases posing a serious health hazards. In Indian coal mines 75% (Singh et.al., 2004) of the coal fires occur due to spontaneous combustion. The main aspect of starting the fire in India is that the coal seams are thicker and there is a tendency of spontaneous heating during the depillaring operation. The problem of extraction of thick seam and coal standing in pillars is a serious one particularly in cases where they are with high moisture, high volatile and low ash content which are more liable to spontaneous combustion. It is not practicable to extract all the coal by caving method or even by complete packing under Indian mining condition. Pillars standing for long time are liable to deteriorate in straight and spilling may occur.

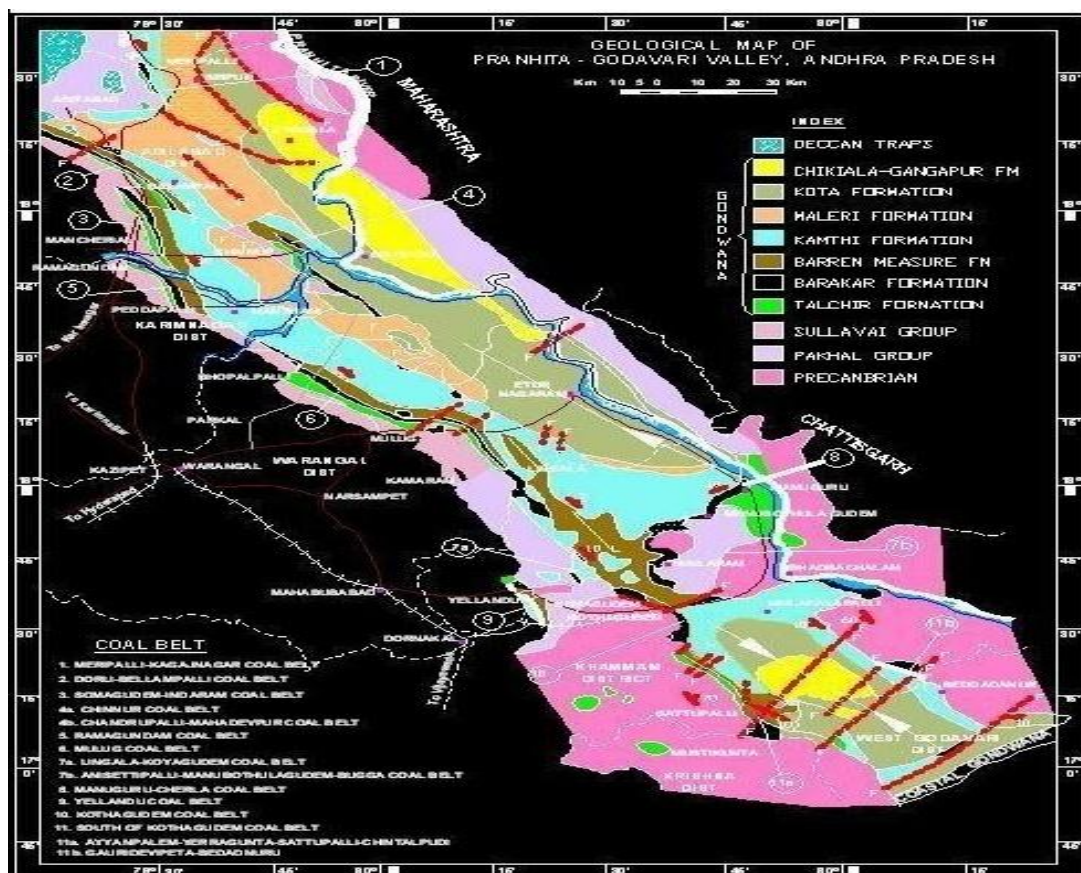


Plate 2.2 Area map of coal fields of SCCL

2.3 Spontaneous heating

The phenomenon in which the coal catches fire automatically on coming in contact with oxygen in the atmosphere without any external source of fire which leads to mine fires is known as spontaneous heating of coal. It is primarily thought that the main cause of spontaneous heating is the self oxidation of coal. Although the actual mechanism of coal oxidation is yet unknown, there are many theories put forward for explanation of the coal oxidation and combustion.

2.4 Mechanism of spontaneous heating

The oxidation of coal, like all oxidation reactions, is exothermic in character. The exact mechanism of the reaction is still not well understood. However, scientists agree that the nature of the interaction between coal and oxygen at very low temperatures is fully physical (adsorption) and changes into a chemisorption form starting from an ambient temperature (Munzner and Peters, 1965; Banerjee, 1985 and Postrzednik et.al., 1988). When coal is exposed to air it absorbs oxygen at the exposed surface. Some fraction of the exposed coal substance absorbs oxygen at a faster rate than others and the oxidation results in the formation of gases. Mainly CO, CO₂, water vapor along with the evolution of heat during the chemical reaction. The rate of oxygen consumption is extremely high during the first few days (particularly the first few hours) following the exposure of a fresh coal surface to the atmosphere. It then decreases very slowly without causing problems unless generated heat is allowed to accumulate in the environment. Under certain conditions, the accumulation of heat cannot be prevented, and with sufficient oxygen (air) supply, the process may reach higher stages. The loose coal-oxygen-water complex formed during the initial stage (peroxy-complexes) decomposes above 70-85⁰ C, yielding CO, CO₂ and H₂O molecules. The rate of chemical reactions and exothermicity change with the rise in temperature, and radical changes take place, starting at about 100⁰ C, mainly due to loss of moisture (Oresko, 1959; Banerjee, 1985 and Handa et.al., 1985). This process continues with the rise in temperature, yielding more stable coal-oxygen complexes until the critical temperature is reached. The ignition temperature of bituminous coal is nearly 160-170⁰ C and of anthracite coal nearly 185⁰ C. Once the coal reaches its ignition point, the air supply to it will only increase the combustion.

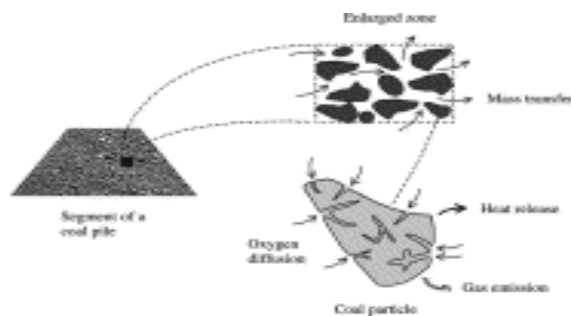


Fig 2.1 Mechanism of spontaneous heating (Weng, 1957).

2.5 Theories of spontaneous heating

2.5.1 Coal oxidation theory (Banerjee et.al., 1985) – various stages of coal oxidation is given in the flow chart below

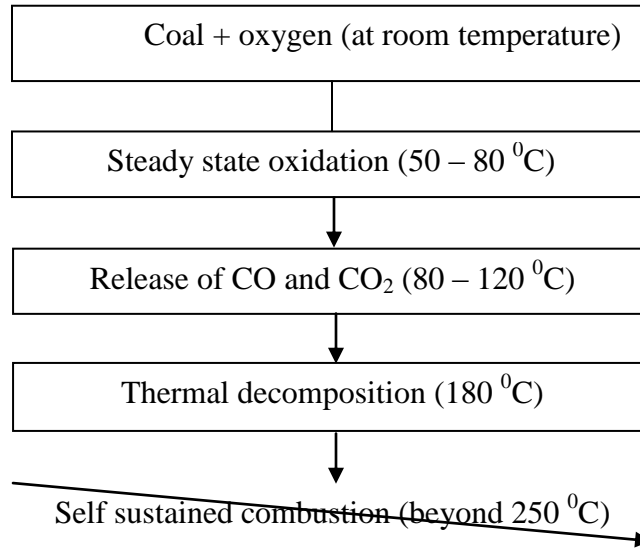


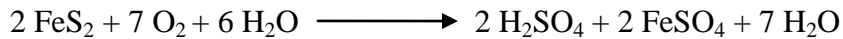
Fig 2.2: Stages of coal oxidation

The overall oxidation process of coal depends on the following factors:

- Temperature – usually rate of chemical reaction increases with temperature rise and is almost doubled for 10°C rise in temperature. The minimum temperature for coal-oxygen reaction is -80°C during which it is physical adsorption and at room temperatures it is chemisorption.
- Type of coal – the intrinsic oxidation mechanism is same for every type of coal but it is the availability of active centers in coal that defines the proneness of a particular coal to spontaneous heating. Usually high moisture and low rank coal have higher oxygen avidity with better ease of peroxy-complex formation and hence higher tendency towards spontaneous heating.
- Extent of oxidation – as the time of exposure of coal surface to air increases the oxidation reaction gradually decreases and the coal gets weathered. In the initial stage the macro pores on surface determine oxygen consumption whereas in later stages the micro pores determine.
- Moisture – moisture adds to heat required for spontaneous combustion by heat of wetting released. It helps in the formation of peroxy complex and influences the rate of reaction. Release of moisture from coal produces more active centers making it more potent to oxidation.

2.5.2 Pyrite theory (Banerjee et.al., 1985) –

The pyrites present as impurities in coal acts as a major source of heat. The oxidation of pyrites is given by the following reaction:



The above reaction is exothermic and it produces heat which is capable enough of triggering spontaneous combustion. Moreover the products of the above reaction have greater amount of volume and hence break open the surface they are embedded in. But it has been found that pyrites can be the cause of spontaneous heating only when they are present in considerable proportions.

2.5.3 Bacterial theory (Jain, 2009) –

Spontaneous heating observed in haystacks and in wood are known to be mainly due to bacterial action. Different evidences showed that bacteria were capable of living on coal and in some cases such bacteria caused a slight rise in temperature of the coal. Graham observed that sterilized coal oxidized at the same rate as the unsterilized coal and concluded that mechanism of oxidation did not include bacterial activity. Fuchs however concluded that bacteria could cause only a slight heating which may not play any significant role.

2.5.4 Phenol theory (Jain, 2009) –

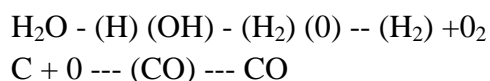
Experiments have shown that phenolic hydroxyls and poly phenols oxidize faster than many other groups. This theory is interesting because it offers a method of determining liability of coal to spontaneous heating.

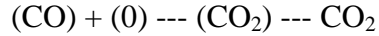
2.5.5 Electro chemical theory (Jain, 2009) –

It explains auto-oxidation of coals as oxidation-reduction processes in micro galvanic cells formed by the coal components.

2.5.6 Humidity theory (Jain, 2009) –

Quantity of heat liberated by atmospheric oxidation of coal is much less than the quantity of heat required removing water from the coal. Thus it can be concluded that if the evaporation of water can be induced at the seat of heating, then the temperature of heating would decrease. Mukherjee and Lahiri (1957) proposed the following mechanism of the reaction between water and coal at 100°C. (Brackets indicate chemisorptions):





When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above scheme well explains other possible sources of CO and CO₂ in low temperature reaction between coal and oxygen.

2.6 Factors affecting spontaneous heating

The main reason for the difficulties in understanding the mechanism of spontaneous combustion is the presence of many internal and external factors affecting the initiation and development of the phenomenon. These factors have been reviewed by various researchers (Kröger and Beier, 1962; Guney, 1968; Chamberlain and Hall, 1973; Feng et al., 1973; Beier, 1973; Kim, 1977; Banerjee, 1982; Didari, 1988; Goodarzi and Gentzis, 1991; Didari and Ökten, 1994).

The main factors which have significant effects on the process are summarized below:

2.6.1 Intrinsic factors – These factors are mainly related to nature of coal

- Pyrites – As pyrite content increases the tendency of spontaneous heating increases.
- Inherent moisture – Changes in moisture content such as drying or wetting of coal have significant effects.
- Particle size and surface area – As particle size decreases the exposed surface area increases and the susceptibility increases.
- Rank and Petrographic constituents – Lower rank coals are more susceptible.
- Chemical constituents – Ash generally decreases liability for spontaneous heating but certain parts of ash such as lime, soda; iron compounds have accelerating effect whereas alumina and silica have retarding effects.
- Mineral matter – Some chemicals promote and others inhibit spontaneous heating.

2.6.2 Extrinsic factors – These factors are mainly related to atmospheric, mining and geological conditions

- Temperature – Higher surrounding temperature leads to increase in oxidation process and ultimately in spontaneous heating of coal.
- Extraneous moisture – Evaporation of surface moisture leads to release of heat of wetting which adds to the temperature rise and increases susceptibility. Presence of atmospheric moisture increases rate of oxidation of coal.
- Oxygen concentration – Higher the oxygen concentration in the atmosphere more rapid is the oxidation process as oxygen is readily available.
- Coal seam and surrounding strata – Presence of faults that lead to the passage of air and oxygen to the heating are generally increase the rate of heating.
- Method of working, ventilation and air flow rate – Mining methods such as bord and pillar mining that leave some pillars attracts more spontaneous heating than longwall methods. Air flow rate controls heating to a large extent. If the ideal flow rate is maintained then it helps in dissipation of heat but if too much of air is flowing then it stagnates the heat and increases the heating.
- Timbering, roadways, bacteria and barometric pressure – Presence of timbers in the mines leads to the danger of catching of fire which gives the heat required for spontaneous heating of coal. Bacterial decomposition of coal and other wood products also releases some amount of heat which increases susceptibility.

The following table gives a summary of all the factors that affect spontaneous heating tendency of coal.

Table 2.1 Set elements of mining conditions (Banerjee, 1982)

Sl. No.	Mining parameter / conditions	Set elements	Probability of spontaneous fire risk	
			High	Low
1.	Category of coal (Chemical nature)	a) Highly susceptible b) Poorly susceptible	High -	- Low
2.	Friability of coal	a) Highly friable b) Poor friability	High -	- Low
3.	Method of working	a) Bord and Pillar b) Longwall	High -	- Low
4.	State of stowing	a) Extraction with caving b) With complete stowing	High -	- Low
5.	Seam thickness	a) High (>5m) b) Low (<4m)	High -	- Low

6.	State of extraction	a) Partial extraction b) Complete extraction	High -	- Low
7.	Nature of extraction	a) Extraction with more than one slice b) In one slice	High -	- Low
8.	Geological disturbances	a) Present b) Absent	High -	- Low
9.	Rock bumps	a) Present b) Absent	High -	- Low
10.	Dykes	a) Present b) Absent	High -	- Low
11.	Overburden	a) Greater than 300m b) Less than 300m	High -	- Low
12.	Parting	a) Shale structure b) Rocky and consolidated	High -	- Low
13.	State of consolidation of barrier	a) Fractured and crushed b) Well consolidated	High -	- Low
14.	Scope of accumulation of fines	a) Fine accumulation sustained b) Fines avoided	High -	- Low
15.	Method of ventilation	a) Advancing type b) Retreating type	High -	- Low
16.	Quantity of ventilation	a) Intensity of pressure difference high b) Low pressure difference	High -	- Low
17.	Humidity	a) Wet mines b) Dry	High -	- Low
18.	Source of hot spots	a) Present b) Absent	High -	- Low
19.	Gas emission rate	a) Low b) High	High -	- Low
20.	Size of panel of the face	a) Large b) Small	High -	- Low
21.	Rate of face advance	a) Slow b) Fast	High -	- Low
22.	Chances for blockage of face advance	a) Present b) Absent	High -	- Low

2.7 National and International status

National status:

Bhattacharya (1971) carried out laboratory experiments to measure the rates of heat release from different coals by a calorimeter during sorption of water vapour in isothermal conditions. It was observed that the rate of heat generation in a particular coal increases with the equilibrium

humidity deficiency of the coal, i.e. with the difference of equilibrium humidity of air and coal. For a given coal, the rate of heat generation due to oxidation has been found to be negligible in comparison with that due to sorption of water vapour. A small 'peak' at the beginning of the rate curves has been observed during the tests with dry coals, with the exception of anthracite; explanations for this phenomenon have been attempted. The results also show that under a given test condition the characteristic rate of heat release is dependent on the type of coal, its particle size and its weathering.

Mishra et.al. (1980) presented a critical analysis of various genetic and physical factors associated with certain Early and Late Permian and Oligocene coal seams and early Eocene lignite seams of India revealed that the high secondary porosity and small particle size, irrespective of petrographic and rank properties in sub-humid climate, favour spontaneous combustion. Large particle size, high rates of gas emission and highly wet or dry seams, in combination with each other tend to inhibit spontaneous heating in sub-humid, per-humid and semi-arid conditions. The susceptibility to auto-ignition of a coal or lignite seam is not uniform throughout its lateral extent and a high inherent porosity at any given rank stage and a high content of susceptible or oxidizable constituents are not sufficient to cause auto-ignition.

Ghosh (1985) made an attempt to evolve a method to identify coal's proneness to spontaneous combustion. It has been shown that if pyrite is present in a coal in finely divided form, the proneness of coals towards spontaneous combustion increases; and the temperature of a coal bed increases if water is added to it, which tends to indicate that water spraying or even flooding cannot be considered as an effective measure to control spontaneous combustion. Moreover, it was also suggested that if a coal body is chilled (to -193°C) the micropores and microcracks in the coal are possibly contracted. Atmospheric oxygen is less likely to enter the coal through micropores and microcracks; and hence chances of spontaneous combustion due to auto-oxidation are diminished.

Tarafdar et.al. (1987) reported results of wet oxidation of coal using alkaline permanganate solution involving measurements of differential temperature at different temperatures, at a constant heating rate, and potential changes between a saturated calomel electrode and a carbon electrode immersed in the coal oxidant mixture within a definite reaction time at a constant temperature. The measurements were made on seven coal samples coalfield of known crossing point temperatures (CPT). Four samples, considered to be highly susceptible to spontaneous heating, had CPT in the range $132-137^{\circ}\text{C}$, and three, considered poorly susceptible to spontaneous heating, had CPT values in the range $162-168^{\circ}\text{C}$, showing two distinct zones of correlation between CPT values and the corresponding differential peak temperatures, and between CPT and the observed potential changes. It was suggested that differential temperature and potential difference measurements during wet oxidation of coal may be used as alternative techniques for the assessment of tendency to spontaneous heating.

Chandra et.al. (1990) conducted a preliminary survey of the frequency of occurrence of fire due to spontaneous combustion in the different seams of the Raniganj Coalfield and showed the possibility of a relationship between coalification and spontaneous combustion of coals. Besides rank, as evidenced from reflectance studies, the amount of vitrinite and exinite contents of the coal seams also influenced the spontaneous combustibility of the coal seams. They found that pyrite of the Raniganj Coalfield had no influence on the combustibility of the coal seams. It was concluded that the proneness to spontaneous combustion of the coals is related to coalification. As the coalification increases, the intensity of spontaneous combustibility decreases gradually from highly susceptible to moderately susceptible to least susceptible to the spontaneous combustion stage.

Bhatt et.al. (1995) developed a reaction-diffusion approach to consider the effects of moisture evaporation and condensation on the rate of oxidation of coal. For a single isothermal particle, pseudo-steady-state balances on moisture and oxygen permit calculation of the effect of different levels of coal-bound moisture on the rate of oxidation. It was shown that partial wetting of coal or condensation of moisture exerts two competing influences on the overall rate of oxidation. On the one hand, a portion of the coal fills up with liquid moisture; in this region, the rate of oxidation becomes negligible, since the oxygen has to dissolve in the moisture before it can gain access to an active site on the coal surface and the solubility of oxygen in water is low. On the other hand, condensation also leads to the release of the latent heat of vaporization. This heat effect raises the temperature of the particle and increases the rate of oxidation in the dry region of the coal. The relative magnitude of the rates of these competing influences determines whether the potential for spontaneous combustion is abated or enhanced..

Sahu, et al. (2004) presented the method of finding out the spontaneous heating susceptibility of coal samples by using differential scanning calorimetry (DSC). 30 coal samples collected from 7 different Indian coalfields have been studied by this method and the onset temperature for all the samples were determined. In addition, the crossing point temperatures (CPT) of all the samples were determined and a comparative study between onset temperature and CPT was presented.

Panigrahi et.al. (2004) carried out extensive field studies to investigate the pillar fire problems in one coal mine in India. A Thermal IR gun and a Thermo vision camera have been employed for thermal scanning to assess the state of heating in selected pillars. Special sampling setups have been designed to collect gas samples from the holes drilled into the selected pillars and the multi gas detector is used to analyse the composition of samples in situ. In order to predict the spontaneous heating in coal pillars, different gas ratios have been calculated and it has been observed that some of the established gas ratios, viz. Graham's ratio, Young's ratio etc. have resulted in negative values in samples containing high amounts of methane. The modified gas ratios have been proposed which will be useful for predicting the pillar fires. These ratios may also be used for assessing the condition of fires in sealed-off areas.

Singh, et al. (2004) observed that exploitation of coal seams from underground mines has become a major challenge to Indian coal mining industries. The problem of spontaneous heating in blasting gallery (BG) panels during extraction is a major threat to safety and productivity in SCCL mines. Most of the BG panels have been sealed due to the occurrences of spontaneous heating during extraction of the panel. After sealing of the panel, it is much difficult to re-open the panel.

Sahu, et al. (2005) described the determination of spontaneous heating susceptibility of coal samples by three different experimental methods viz., crossing point temperature (CPT), differential thermal analysis (DTA) and differential scanning calorimetry (DSC) since all coals are not susceptible to spontaneous heating to the same extent, it was essential to assess their degree of proneness in order to plan advance precautionary measures.. The acceptability of a method for determining spontaneous heating characteristics of coal mainly depends upon how closely it predicts the spontaneous heating behaviour in the field conditions. Considering this, it may be concluded that the onset temperature obtained from differential scanning calorimetry may be a better method than crossing point temperature.

Singh, et al. (2007) observed in opencast mines, coal immediately oxidises and catches fire due to the intrinsic characteristics of coal, such as low rank, high moisture, high volatile matter, presence of sulfur in the form of pyrites, low crossing point temperature (CPT) and ignition point temperature (IPT) value and less incubation period. In opencast mines, when the coal benches are left idle for a longer time, heat accumulation takes place in favourable conditions and sometimes leads to fire. The purpose of this paper is to present the different successful case studies regarding the safety management of open pit coal mines from occurrences of spontaneous heating.

Ahmed, et al. (2008) calculated Liability index using CPT for studying the propensity of coal towards spontaneous heating. CPT that has been in lab requires extra precaution for repeatable results. To overcome the difficulties, attempts were made to study the relationship between peripheral oxygen groups or the functional oxygen groups in coals and their correlation with proneness to auto-oxidation using liability index (LI). Also the correlation between liability index and crossing point temperature have been presented in this paper.

Sahay et.al. (2008) proposed realistic characterisation of coal towards spontaneous heating for taking corrective measures. They developed a methodology based on thermo-decompositional study of coal sample for determination of minimum temperature at which coal bed temperature starts self propellant known as critical temperature and a new liability index based on it. They presented a brief description of different liability index particularly based on thermal study, a critical analysis of dependency of critical temperature on moisture content, ash content, volatile matter, carbon content, surface area and porosity, new liability index based on

thermodecompositional study of coal sample and correlation with liability index model based on coal proximate analysis results of coal sample including surface area and porosity.

Mohalik, et al. (2009) presented the review of application of three thermal techniques viz; differential thermal analysis (DTA), thermogravimetry (TG) and differential scanning calorimetry (DSC); for studying the susceptibility of coal to spontaneous heating and fire. It also critically analyses the experimental standards adopted by different researchers, while applying these techniques in studying thermal behaviour of coal samples. The paper also presents the future direction of research in this subject area.

Nimaje et.al. (2010) made thermal studies on spontaneous heating of coal. Of all the experimental techniques developed thermal studies play an important and dominant role in assessing the spontaneous heating susceptibility of coal. They made an overview of thermal studies carried out by different researchers across the globe for determination of spontaneous heating of coal and revealed that lot of emphasis on experimental techniques is necessary for evolving appropriate strategies and effective plans in advance to prevent occurrence and spread of fire.

International status:

Peter, et al. (1978) observed the oxidation of the weathered materials has an apparent activation energy lying between 63.9 and 69.0 kJ/mol which is independent of their moisture content. However, the rate of oxidation of char increases with increasing moisture content and decreases with increasing carbonization temperature of the parent coal, and with the extent of the char's weathering.

Singh, et al. (1984) outlined the current techniques of assessment of spontaneous combustion risk indices for classifying coal seams liable to self-heating. Factors affecting liability of coal to spontaneous combustion depend upon intrinsic factors as well as external factors promoting the self-heating. An adiabatic oxidation test was described which can be used to assess the liability of coal according to intrinsic reactivity. Systems of risk classifications are based on the synthesis of ratings assigned to intrinsic as well as extrinsic factors. Precautionary measures to control spontaneous combustion hazard in underground longwall mining, stockpiling and seaborne transport of coal are described together with the techniques of fire-fighting using liquid nitrogen.

Gouws et.al. (1988) gave three characteristics on a differential thermal analysis thermogram (i.e., the crossing-point temperature, stage II exothermicity gradient and the transition temperature to high-level exothermicity) are generally believed to be indicative of the self-heating propensity of coal. A new index was developed and applied to 58 coals, enabling known dangerous and safe coals to be identified.

Olayinka, et al. (1990) showed the CPT of Nigerian coals were found to decrease with increase in coal rank. The liability index, which gives a better evaluation of susceptibility of coal to spontaneous heating, was also found to decrease with increase in rank and with decrease in oxygen content and moisture holding capacity of the coal. Of the four coals studied, the high volatile bituminous coal had the lowest susceptibility to spontaneous combustion while the subbituminous was the most susceptible.

Gouws, et al. (1990) designed an adiabatic calorimeter to enable the spontaneous combustion propensity of coal to be established. Various indicators of self-heating potential, such as total temperature rise, initial rate of heating, minimum self-heating temperature, and kinetic constants were investigated. Results obtained from the adiabatic tests were compared with the results of crossing-point temperature determinations and differential thermal analysis (DTA) tests for the same coals, with a view to formulating a mathematically consistent spontaneous combustion liability index. This paper describes the major components of the adiabatic calorimeter.

Anthony, et al. (1995) said that self-heating of coal mainly involves exothermic reactions of oxygen at reactive radical sites within the coal and the enhancing or moderating effect that water had on these reactions. The thermal response of samples of low-rank coals, dried by heating under nitrogen flow at 105°C and exposed to dry oxygen, is similar to or slightly less than that observed when they are flow-dried at 30°C and tightly bound moisture remains. The most likely reason is that moisture affects the nature of the radical sites where oxidation occurs. By hindering the formation of stabilized radicals, it encourages faster oxidation which may lead to enhanced thermal response, although some of the extra heat may be taken up by the residual moisture. When loosely bound moisture is allowed to remain in the coal, the thermal response on exposure to dry oxygen decreases very quickly, due mainly to hindered access to reactive sites and dissipation of heat generated by any oxidation that does occur. The effect of desorption is comparatively minor and the course of the oxidation reaction responsible for generating heat does not appear to be changed by the presence of small quantities of loosely bound moisture.

Jose, et al. (1995) used differential thermal analysis (DTA) as a method to study the self-heating behaviour of fresh and oxidized coals. Oxidation was performed in air at 200°C for periods of up to 72 h. As the rank of the coal increases, both the self-heating and the end of combustion temperatures also increase. The total heat loss (area under the DTA curve) increases with the rank of the coal. An increase in the self-heating temperature, a decrease in the temperature of the end of combustion and a decrease in total heat flow were observed as a consequence of coal oxidation. A relationship between the total heat loss and the calorific value as determined using the ASTM standard method is pointed out.

Vancea, et al. (1995) work investigates the effect of the moisture content of coal on its spontaneous ignition in oxygen (40°C–140°C). It has been found that the highest heating rate is

achieved at a medium moisture content of 7 wt% for an initial inherent moisture content of the coal before drying (in dry nitrogen at 65°C) of 20 wt%. This is particularly noticeable at temperatures below 80°C and tends to support previous studies showing that a maximum oxidation rate occurs at such moisture content in the same temperature range.

Ren, et al. (1998) used adiabatic calorimeter for the propensity of 18 pulverised coals (Australia, UK, US, Indonesia, South Africa, South America) to spontaneous combustion. All the coal samples were tested at an initial temperature of 40°C and three samples at 60°C. Their propensities to spontaneous combustion were ranked according to their initial rate of heating (IRH) and total temperature rise (TTR) values. The results demonstrated that air humidity is an important factor is determined whether a heating will progress rapidly or not. The particle size distribution of the coal affects the IRH and TTR values, with relatively smaller particles tending to be more reactive. Aged and pre-oxidised coals have higher IRH and lower TTR values, and the coal becomes less reactive. The magnitude of the temperature raises (TTR) increases with increasing initial temperature.

Rosema et.al. (2000) developed a numerical simulation model “COALTEMP” to study the oxidation and possible spontaneous combustion of coal that is exposed to the atmosphere and the daily cycle of solar irradiation. First the differential equations that describe heat flow, oxygen flow and oxidation in the coal matrix, and the equations describing the exchange of heat, radiation and oxygen with the atmosphere, are presented.

Kuçuka, et al. (2001) evaluated the spontaneous combustion characteristics of Askale lignite from Turkey. The effect of the gas flow rate, the moisture of the piles of coal, the humidity of the air and particle size on the spontaneous combustion characteristics of coal samples were examined using Crossing Point Methods adapted to their laboratories conditions. The liability of spontaneous combustion of this lignite was found to increase with decreasing particle size, increasing moisture content of the coal and decreasing humidity of the air.

Yucel et.al. (2001) conducted an experimental study aimed at evaluating the spontaneous combustion characteristics of two Turkish lignites moistured and air-dried at varying times. The content of three predominant oxygen functional groups (carboxyl, hydroxyl, and carbonyl) of untreated, moisten and air-dried coal samples were also determined with wet chemical methods. The content of oxygen functional groups in moisten coal samples do not differ significantly that of untreated coal samples, for realized in vacuum dessicator to moisture of coal samples. The liability of spontaneous combustion of the two coals were found to reduce when moisture content increased with increase in contacted time to water vapour.

Nelson et.al. (2007) used a wide variety of techniques to gain insight into the processes that govern the self-heating of coal. These include oxidation mechanisms, ranking the propensity of different coals to self-heat, and the detection and suppression of self-heating. Moist coal in coal

mines and stockpiles have very different combustion characteristics than those predicted on the basis of dry testing. Consequently, methods for ranking the propensity of coal to spontaneously combust in actual mining conditions need to be developed.

Daiyong et.al. (2007) assumed that spontaneous combustion of coal seams is a complicated process that is a function of the interplay of internal and external conditions. Based on geologic field investigations and comprehensive analyses, four models of spontaneous combustion for coal were established: A genesis-type model, a coal-fires propagation model, a model for the progressive stages and products of a coal fire and a cross-sectional model of zones.

Table 2.2: Experimental parameters used by different researchers in DTA studies on spontaneous heating of coal (Mohalik et al, 2009)

Sl No	Name of Author	Year	Parameters							
			Particle size/ mesh	Heating rate °C/ min	Atm.	Sample Amt. (mg)	Flow rate mL/ min	No. of sample studied	Ref. material	Temp. range /°C
01	Whitehead and Breger	1950	-	10,20	Air/ Vacuum	-	-	-	-	Amb. to 550
02	Glass	1955	-100	10,20	-	-	-	7	-	Amb. to 1000
03	Berkowitz	1957	-65	6	N ₂	100	2.5	6	Dry quartz	Amb. to 500
04	Banerjee and Chakraborty	1967	-72, -200, -10 +60	1,3,5,10, 15	Atm. Air	600	-	6	Calcined alumina	Amb. to 400
05	Banerjee et al.	1972	-72	5	Atm. Air	600	-	6	Calcined alumina	Amb. to 400
06	Haykiri-Acma et al.	1993	-	15	N ₂	-	-	-	-	Amb. to 1000
07	Podder et al.	1995	-100	10	Ar	10	100	5	-	30 - 900
08	Iordanidis et al.	2001	-16	10	N ₂	-	150	7	Alumina	Amb. to 1000
09	Kok	2002	-60	10	Air	10	167	4	Alumina	20 - 900
10	Panigrahi, Sahu	2004	-72	30	Atm. Air	600	-	31	Alpha Alumina	Amb. to 400
11	Elbeyli and Piskin	2006	-65	10	Air/ N ₂	10	100	1	-	Amb. to 1000
12	Haykiri-Acma et al.	2006	-65	20	Atm.	20	-	7	Alumina	Amb. to 1000
13	Sis	2007	-8+10 to -400	10	Air	10	50	1	Alpha Alumina	Amb. to 900
14	Ozbas	2008	-	10	N ₂	10	50	15	Alpha Alumina	Amb. to 900

Table 2.3: Experimental parameters used by different researchers in TG studies on spontaneous heating of coal (Mohalik et al, 2009)

Sl No	Name of Author	Year	Parameters							
			Particle size/ Mesh	Heating rate/ °C /min	Atm.	Sample amount / mg	Flow rate mL/ min	No. of sample	Ref. material	Temp range /°C
01	Ciuryla and Welmar	1979	-	20	Helium	-	1000	4	-	Amb. to 1000
02	Cumming	1980	-	15	Air	20	75	22	-	Amb. to 900
03	Smith and Neavel	1981	-	15	Air	300	-	66	-	Amb. to 1000
04	Pranda et al.	1999	-72	5	-	10	-	1	-	200-700
05	Podder and Majumder	2001	-100	10	N ₂	10	100	5	-	30-900
06	Alonso et al.	2001	-400	25	Air	13	50	10	-	Amb. to 1000
07	Kok and Keskin	2001	-60	10	N ₂ Air	10	50	10	-	20-800
08	Kok	2002	-60	10	Air	10	167	4	-	20-900
09	Avid	2002	-	10,20,30,40,50	N ₂ CO ₂	500			-	Amb. to 1000
10	Ozbas et al.	2002	-60	10	Air	10	50	4	-	20-900
11	Ozbas et al.	2003	-60	5,10,15,20	Air	10	50	4	-	20-900
12	Kizgut et al.	2003	-200	10	N ₂	10	15	7	-	Amb. to 700
13	Sonibare et al.	2005	-100	10	Air N ₂	10,15	50	5	-	25-1000
14	Umar et al.	2005	-200	10	Air	500	25	2	-	Amb. to 800
15	Gunes et al.	2005		20	N ₂		250	12	-	140-900
16	Kok	2005	-60	10	Air	10	50	17	-	20-600
17	Wachowski and Hofman	2006	-170	3	Helium	10	-	4	-	20-1000
18	Mianowski et al.	2006		30	Air	500	-	4	-	Amb. to 900
19	Haykiri-Acma et al.	2006	-65	20	Air	20	-	7	-	Amb. to 1000
20	Sis	2007	-8+10 to -400	10	Air	10	50	1	Alumina	Amb. to 900
21	Sensogut et al.	2008	-150	10	N ₂	100		10	Alpha Alumina	Amb. to 1600
22	Ozbas	2008	-	10	N ₂	10	50	15	Alpha Alumina	Amb. to 900

CHAPTER – 3

EXPERIMENTAL TECHNIQUES

To study the effects of various parameters of coal that affect the spontaneous heating tendency of coal, the following experiments are needed to be carried out:

- Proximate analysis
- Ultimate analysis
- Petrographic analysis
- Calorific value
- Differential thermal analysis
- Differential scanning calorimetry
- Crossing point temperature
- Wet oxidation potential
- Flammability temperature
- Critical air blast
- Olpinski index

3.1 SAMPLE COLLECTION AND PREPARATION

It is the process by which the physical and chemical properties of the mineral or ore can be ascertained with the desired accuracy. It is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole.

Different types of sampling are:

- Channel sampling
- Chip sampling
- Grab sampling
- Bulk sampling
- Drill hole sampling

Chip sampling is done in hard ores where it is difficult to cut the channels. It can be taken in case of uniform ores and where the rock structures are independent of the values. The sample is collected by breaking of small equal sized chips from a face at points usually equally spaced both vertically and horizontally.

Grab sampling is applied to the broken ore in the stope or at the face, ore transported. Usually grab sampling of the ore broken in the stope is unreliable as accurate estimation of the volume of broken ore is impossible. Grab sampling of tubs or ships is however more representations since samples are collected from units of regular volume.

Bulk sampling is done where conventional sampling methods do not give a representative scale; large scale sampling or bulk sampling resorted to. Bulk samples eliminate the effect of irregular distribution of value or minor.

For our Project work, channel sampling method has been carried out which is common among various techniques discussed above.

3.1.1 Channel sampling (IS 436 Part I/Section I - 1964)

The section of seam to be sampled shall be exposed from the roof to the floor. The seam sample shall, be taken in a channel representing the entire cross-section of the seam having the dimensions of 30 x 10 cm, that is, 30 cm in width and 10 cm in depth. For this purpose, two parallel lines, 30 cm apart end at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, freshly exposed surface of the seam. Obvious dirt bands exceeding 10 cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimized.

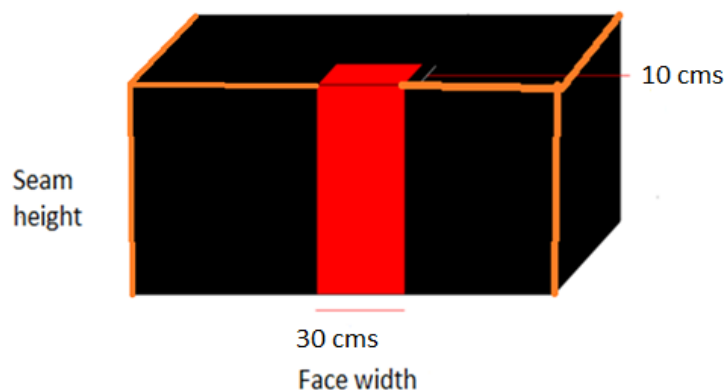


Fig 3.1 Channel Sampling

3.1.2 Sample preparation (IS: 436 (Part 1/Section 1)-1964 and IS: 436 (Part II)-1965)

The samples received from the field via channel sampling are crushed in the laboratory as per the experimental requirements. The crushed sample is then sieved to required sizes and stored in air

tight polythene packets. The packets are stored in air tight containers for further use in experimentation.

3.2 METHODS FOR DETERMINING INTRINSIC PROPERTIES OF COAL

3.2.1 Proximate analysis (IS 1350 Part I -1984)

The objective of coal ultimate analysis is to determine the amount of fixed carbon (FC), volatile matters (VM), moisture, and ash within the coal sample. The variables are measured in weight percent (wt. %) and are calculated on different basis.

- Ar (ash-received) basis – puts all variables into consideration and uses the total weight as the basis of measurement.
- Ad (air-dried) basis – neglects the presence of moistures other than inherent moisture.
- db (dry-basis) basis – leaves out all moistures, including surface moisture, inherent moisture, and other moistures.
- daf (dry, ash free) basis – neglects all moisture and ash constituent in coal.
- dmmf (dry, mineral-matter-free) basis – leaves out the presence of moisture and mineral matters in coal.

3.2.1.1 Moisture

Procedure:

- Take 1 g of – 212 micron (BSS) coal sample in a glass crucible.
- Put it in a furnace at 110°C for 90 minutes.
- Remove the sample after 90 minutes and weigh the glass crucible again.
- Calculate the moisture content by the formula

Total moisture content of the original sample, $A = (X - Y)/X * 100$

Where,

X – Initial mass of the coal sample before heating

Y – Final mass of coal sample after heating

3.2.1.2 Ash

Procedure:

- Take 1g of -212 micron (BSS) coal sample in a silica crucible.

- Heat the sample in a muffle furnace at 450⁰ C for 30 minutes and then further heat it for 1 hour with temperature rising from 450 to 850⁰ C.
- Remove the silica crucible and then allow it to cool in a dessicator for 15 minutes and weigh the crucible again.
- Calculate ash content by the formula

$$\text{Ash percentage, } A = (100 * (M_3 - M_4)) / (M_2 - M_1)$$

Where,

M₁ – Mass of crucible (g)

M₂ – Mass of crucible and sample

M₃ – Mass of crucible and ash

M₄ – Mass of the crucible after brushing out the ash and reweighing it

3.2.1.3 Volatile matter

Procedure:

- Take 1 g of -212 micron coal sample in a crucible and put the lid.
- Put the crucible in a furnace maintained at 925⁰ C for 7 minutes exactly.
- Take out the crucible and weigh it again.
- Calculate the volatile matter content by using the relation

$$\text{Volatile matter percentage, } V = (100 * (M_2 - M_3) / (M_2 - M_1)) - M_0$$

Where,

M₀ – Percentage of moisture in the sample on air dried basis

M₁ – Mass of empty crucible and lid

M₂ – Mass of crucible plus lid and sample before heating

M₃ – Mass of crucible plus lid and sample after heating

3.2.1.4 Fixed carbon

It is determined by subtracting the sum of all the above parameters and is given as

$$\text{Fixed Carbon, } FC = 100 - (M + V + A)$$

Where,

M – Moisture content

V – Volatile matter content

A – Ash content

3.2.2 Ultimate analysis (IS 1351 - 1959)

The objective of coal ultimate analysis is to determine the constituent of coal, but rather in a form of its basic chemical elements. The ultimate analysis determines the amount of carbon (C), hydrogen (H), oxygen (O), sulfur (S), and other elements within the coal sample.

3.2.3 Petrographic analysis (IS 9127 Part II - 1992)

Coal is a rock composed of number of distinct organic entities called macerals and lesser known amounts of inorganic substance called as minerals. Each maceral has a distinct set of property and it influences the behavior of coal. Coal surface can be analyzed at macroscopic level and microscopic level. At macroscopic level coal appears as banded or non banded rock. The bands are divided into four major lithotypes – Vitrain, Clarain, Durain, Fusain. At microscopic level coal has three basic groups of macerals and mineral matter. The macerals are of three types – Vitrinite, Liptinite, Inertinite and Visible mineral matter.

3.2.4 Calorific value (IS 1350 – 1959)



Plate 3.1 Bomb calorimeter

The calorific value or heat of combustion or heating value of a sample of fuel is defined as the amount of heat evolved when a unit weight (or volume in the case of a sample of gaseous fuels) of the fuel is completely burnt and the products of combustion cooled to a standard temperature of 298°K.

Procedure:

- About 1 g of coal sample is taken in a pellet press and a pellet is formed of nearly 1 g weight.
- The pellet is put in the lid provided and the nickel wire is put on.
- A thread is suspended from the nickel wire that is in direct contact with the coal pellet.
- The bomb is now closed by putting on the lid firmly from the top.
- Oxygen is supplied into it till the pressure is 30 bar inside the bomb.
- 1 kg of water is put into the vessel provided and the bomb is put in the stand so that the top of the bomb is in layer of water level.
- The overall lid is closed and the stirrer is switched on along with the digital thermometer.
- The stirrer is allowed to run till the temperature attains a near constant value.
- The cord is put into the furnace and the bomb is fired after the attainment of constant value.
- The temperature rises initially at high rate and later settles down at a constant value.
- The initial and final temperature is noted.
- The water equivalent of the instrument is noted.
- The calorific value of coal is calculated by using the relation

$$\text{Calorific value} = ((T_f - T_i) * \text{Water equivalent}) / \text{Weight of the pellet}$$

Where,

T_f – Final temperature

T_i – Initial temperature

$$\text{Useful heat value} = 8900 - 138(A+M)$$

The following grade system is followed in India for grading of non coking coals.

Table 3.1 Grading of non coking coal (IS 1350 - 1959)

Grade	Useful Heat Value (UHV)(Kcal/Kg) UHV= 8900-138(A+M)	Corresponding Ash% + Moisture % at (60% RH & 40^o C)	Gross Calorific Value GCV (Kcal/ Kg) (at 5% moisture level)
A	Exceeding 6200	Not exceeding 19.5	Exceeding 6454
B	Exceeding 5600 but not exceeding 6200	19.6 to 23.8	Exceeding 6049 but not exceeding 6454
C	Exceeding 4940 but not exceeding 5600	23.9 to 28.6	Exceeding 5597 but not exceeding. 6049
D	Exceeding 4200 but not exceeding 4940	28.7 to 34.0	Exceeding 5089 but not Exceeding 5597
E	Exceeding 3360 but not exceeding 4200	34.1 to 40.0	Exceeding 4324 but not exceeding 5089
F	Exceeding 2400 but not exceeding 3360	40.1 to 47.0	Exceeding 3865 but not exceeding. 4324
G	Exceeding 1300 but not exceeding 2400	47.1 to 55.0	Exceeding 3113 but not exceeding 3865

3.3 METHODS FOR DETERMINING SPONTANEOUS HEATING SUSCPETIBILITY OF COAL

3.3.1 Flammability temperature method (Nimaje et al., 2010)

The set up for the determination of the flammability temperature of coal consists of vertical tubular furnace of internal diameter 50mm, length 300mm, open at both ends, a dust dispersing unit, a solenoid valve a reservoir for air, a mercury manometer, a drying tower and an aspirator bulb. Coal dust sample is kept in the helical dust disperser. Air from the reservoir is made to pass through the disperser and on emergency from the divergent mount, forms a uniform dust-air mixture inside the furnace. The minimum temperature at which this mixture catches fire, which is indicated by the appearance of flame coming out of the bottom of the tubular furnace is the flammability temperature of the coal dust.

Procedure:

- Place 200 mg sample of coal having mesh size -200 mesh (BSS) in a helical tube.
- Maintain mercury column difference of 8 cm by aspirator bulb and turn off the tap.

- At desired temperature of furnace, switch on the solenoid valve, which allows the air to pass through it very fast and find out the status of coal sample (spark, smoke or flame).
- If flame appears then find out the exact temperature in lower temperature range by trial and error method, if not then go for higher temperature range.



Plate 3.2 Flammability temperature apparatus

3.3.2 Wet oxidation potential (Singh et.al., 1985, Tarafdar and Guha, 1989, and Panigrahi et.al., 1996)



Plate 3.3 Wet oxidation potential apparatus

It is based on the chemical reaction of coal sample with solution of KMnO_4 and KOH . The solution of alkaline potassium permanganate and potassium hydroxide with coal sample forms an

electrochemical cell which on stirring produces EMF against a standard potential of 0.56V. The plot of the EMF's versus time gives an idea of the susceptibility of the coal sample towards spontaneous heating.

Procedure:

- About 0.5g of -212 micron (BSS) of coal sample is taken in a solution of 100 ml of 1N KMnO_4 and 0.1N of KOH solution.
- The electrodes, both the carbon and saturated calomel electrode, are inserted into the solution.
- The solution is then stirred continuously by using a magnetic stirrer and the EMF readings are noted down.
- The readings are taken at 1 minute time interval up to 30 minutes or till a constant value of EMF is attained.

3.3.3 Crossing point temperature (Didari et.al., 2000)

This is one of the oldest methods for determining susceptibility of coal sample. In this the sample is subjected to uniform constant heating of about 1°C/min and a graph is plotted between temperature of sample and time. The experimental setup consists of an automated oven that is capable of maintaining a constant temperature rise by a programmer. The sample is placed in a tube of 2 cm diameter and length 20 cm. This tube is placed in a glycerine bath and a small tube of 6 mm diameter is wound around the bigger tube to heat the incoming air. About 20 g of coal sample is taken and put over a mesh and glass wool. A thermometer is placed in the bath as well as the tube to measure the respective temperatures.

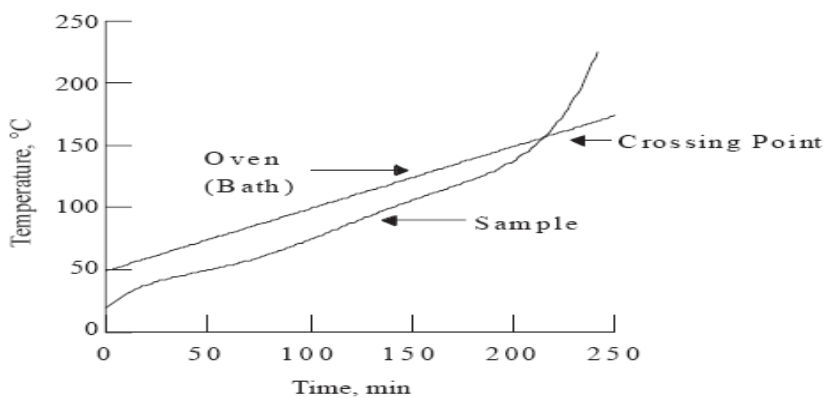


Fig 3.2 CPT curve (Didari et al 2000)

From the graph it can be inferred that initially due to release of moisture from coal sample the temperature decreases but after that the graph is found to be parallel to the constant rate of temperature line for some time. After that the temperature suddenly shoots up and crosses the oven temperature line and rises steeply beyond that. The point on the graph at which the sample temperature cuts the oven temperature is called as Crossing Point Temperature. Lower the crossing point temperature higher is the susceptibility of the coal sample to catch fire.

3.3.4 Olpinski index (Banerjee et.al., 1985) –

In this method a small pellet of coal is allowed to undergo aerial oxidation at a temperature of 235⁰ C. The exothermicity of coal pellet gives the measure of spontaneous heating susceptibility Sz_a of the concerned coal. This method makes correction for ash content of coal (A) and expresses spontaneous heating tendency as

$$Sz_b = Sz_a - (100 / (100 - A))$$

Where

Sz_b – Spontaneous heating index free of ash

Sz_a – Olpinski index

3.3.5 Critical Air Blast (Sahu et.al., 2005) –

Different coals vary in their reactivity to oxygen/ air. Highly reactive coals oxidize faster and this oxidation once started may even be sustained at low temperatures. The reaction of coal with oxygen is termed as oxy-reactivity and this is determined by the critical air blast (CAB) test. The critical air blast is a measure of the reactivity of coal to air. It is the minimum rate of air supply, which maintains combustion of closely graded coal in an ignition bed 'of specified dimensions. The more reactive the coal towards air, lower is its CAB value, and the coal is more susceptible to spontaneous heating.

$$\text{Critical Air Blast (CAB)} = ((1549 * f * V * (P + a - b)) / ((273.15 + T_w) * d))$$

Where,

P – Atmospheric pressure (mm Hg);

V – Air blast rate at which resuscitation occurs (k l/min of dry air);

d – Diameter of combustion chamber (mm);

- f – Gas meter correction factor (1.0);
- a – Manometer pressure (mm Hg);
- h – Aqueous vapour pressure at T C (mm Hg); and

3.3.6 Differential thermal analysis (Nimaje et al., 2010)



Plate 3.4 DTA – TG apparatus

It is often used to determine the physical property of a substance as a function of temperature. This method analyses the effect of temperature on the properties of the sample and compares it with an inert reference material. In DTA the temperature difference between sample and inert reference is measured when both are subjected to identical heat treatments and then plotted against time or temperature. The DTA apparatus consists of a sample and reference holder, a furnace, a temperature programmer to maintain constant temperature rate and an output to monitor the test. The sample holder has two thermocouples each for reference and sample. The sample is contained in a small crucible. The thermocouple should not be in direct contact with sample. By the temperature programmer the temperature is made to rise at a constant rate and the temperature difference of the sample and reference is plotted against time. The plot consists of three parts – stage I, II, III.

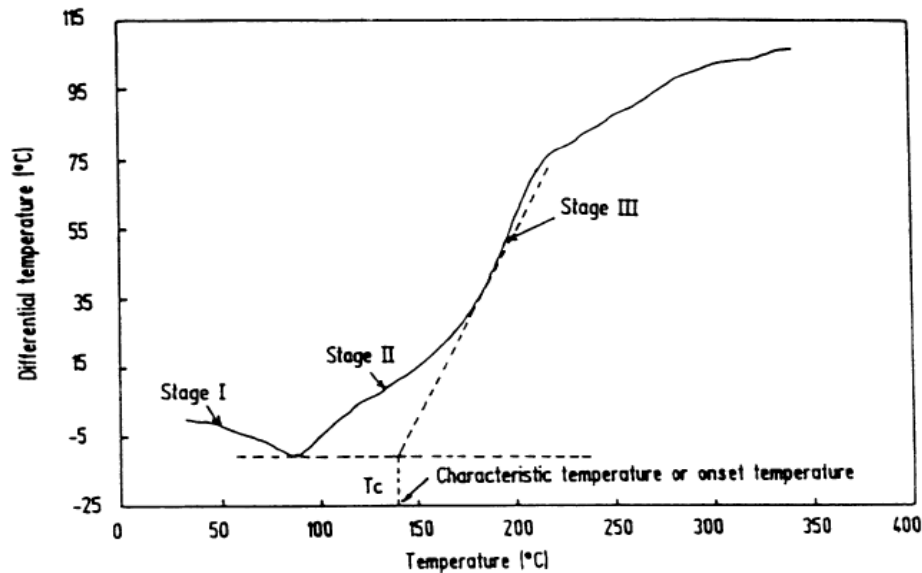


Fig 3.3 Different stages of DTA (Nimaje et al., 2010)

During stage I mostly endothermic reactions take place and the temperature falls due to release of moisture.

Stage II consists of two parts – II A and II B. From the beginning of II A, the heating tendency starts accompanied by a small amount of endothermic reactions. The point where II A begins is called as the inflexion point as the thermogram suddenly rises. In II B exothermic reactions start to dominate.

The beginning of stage III is called as Transition temperature. This temperature is very important as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature the higher is the susceptibility of coal and vice versa. From stage III onwards complete exothermic reactions occur and the temperature continues to rise steeply.

From the thermogram plot four important points are required – slope of II A, II B, overall slope of II and the transition temperature. Lower the slope values lower is the susceptibility. The transition temperature is found out by drawing tangents at the inflexion point and any point on stage III. Their intersection gives the transition temperature.

Procedure:

- About 10 mg of -212 micron (BSS) coal sample is taken in the crucible.
- The reference material taken is alpha alumina.
- The DTA machine is switched on and the software is set according to required conditions viz., heating rate is maintained at 5⁰ C/minute and the final temperature is taken as 450⁰C.

- The plots obtained are then analyzed for the slopes of various stages and transition temperature.

3.3.7 Differential scanning calorimetry (Nimaje et al., 2010, 2010)

It is similar to DTA with the main difference being that in DSC the reference is maintained at a known temperature and while the sample temperature is brought to that temperature the change in energy is recorded. It is more accurate than the DTA. Differential scanning calorimetry (DSC) is used to measure heat flow into or out of a sample as it is exposed to a controlled thermal profile. DSC is a technique in which the ordinate value of an output curve at any given temperature is directly proportional to the differential heat flow between a sample and reference material and in which the area under the measured curve is directly proportional to the total differential calorific input.

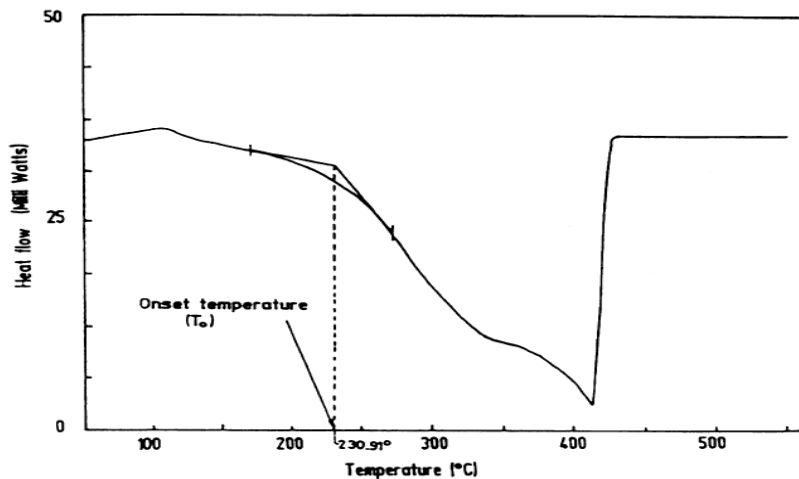


Fig 3.4 Determination of onset temperature of DSC (Nimaje et al., 2010)

It can be observed from the above thermogram that initially the endothermic reaction dominates followed by the exothermic reactions. The temperature of initiation of the exothermic reaction can be considered as an indicator of spontaneous heating susceptibility of coal samples, which is known as the onset temperature. The lower is this temperature, higher is the spontaneous heating susceptibility. To determine the onset temperature or characteristics temperature (T₀) of exothermic reaction, first a tangent is drawn at the inflexion point of the pre-transition. Then a second tangent is drawn at the greatest slope of the first exothermic reaction. The intersection of the two tangents gives the characteristics or onset temperature.

CHAPTER – 4

RESULTS

ABSTRACT OF EXPERIMENTAL TECHNIQUES

1. Proximate analysis

A. Determination of moisture

- Amount of coal : 1 g coal
- Size of coal : - 212 micron (72 mesh)
- Heating time : 1.5 hours at 110⁰ C

B. Determination of volatile matter

- Amount of coal : 1 g of coal
- Size of coal : - 212 micron (72 mesh)
- Heating time : at 900⁰ C for 7 minutes

C. Determination of ash

- Amount of coal : 1 g of coal sample
- Size of coal : - 212 micron (72 mesh)
- Heating time : 30 minutes at 450⁰C and 60 minutes at 850⁰C

2. Wet oxidation potential study

- Amount of coal : 0.5 g of coal sample
- Size of coal : - 212 micron (72 mesh)
- System : Coal + KMnO₄ + KOH

3. Flammability temperature

- Amount of coal : 200 mg of coal sample
- Size of coal : - 72 micron (25 mesh)
- System : Coal + Air
- Volume of air : 500 ml
- Pressure of air : 8 cm of Hg

4. DTA – TG

- Amount of coal : 60 mg of coal sample
- Size of coal : - 212 micron (72 mesh)
- Heating rate : 5⁰ C/min
- System : coal + air

Table 4.1 List of coal samples

Sl. No.	Sample	Name of the organization
1	1	SCCL
2	2	
3	3	
4	4	
5	5	
6	6	
7	7	
8	8	
9	9	

Table 4.2 Results of proximate analysis parameters

Sample	Moisture(M)	Ash(A)		Volatile Matter(VM)			Fixed Carbon(FC)			
	Ar (%)	Ar (%)	Ad (%)	Ar (%)	Ad (%)	daf (%)	Ar (%)	Ad (%)	daf (%)	dmmf (%)
1	2.433	33.07	33.89	27.96	28.65	43.34	36.54	37.45	56.65	76.5
2	2.13	25.94	26.50	33.42	34.14	46.46	38.51	39.34	53.53	58.5
3	2.73	14.46	14.86	35.83	36.83	43.26	46.98	48.29	56.73	76.1
4	3.76	25.68	26.68	34.13	35.46	48.37	36.43	37.85	51.62	70.1
5	3.166	15.28	15.77	35.99	37.16	44.12	45.57	47.05	57.68	76.8
6	3.66	37.84	39.27	25.88	26.86	44.23	32.62	33.85	55.76	68.5
7	3.766	27.15	28.21	32.84	34.12	47.53	36.25	37.66	52.46	55.4
8	3.693	17.41	18.07	40.40	41.94	51.20	38.5	37.97	48.79	81.6
9	2.866	11.04	11.36	38.91	40.05	45.19	47.19	48.57	54.80	60.9

Dry basis – $(100 / 100 - M)$
 Dry ash free basis – $(100 / 100 - (M + A))$
 Dry mineral matter free basis – $(100 / 100 - (M + A + VM))$

Table 4.3 Results of calorific value

Sample	Calorific Value (Kcal/Kg)	UHV Value (Kcal/Kg)	Grade of a Coal
01	3648	4001	E
02	4383	5026	C
03	6479	6527	A
04	4689	4837	D
05	5551	6355	A
06	2853	3173	F
07	4400	4634	D
08	5053	5988	B
09	5533	6981	A

Table 4.4 Results of wet oxidation potential

Sample	Potential difference EMF (mv)
01	143
02	131
03	115
04	134
05	131
06	150
07	149
08	125
09	144

Table 4.5 Results of flammability temperature

Sample	Flammability temperature (°c)
01	520
02	500
03	530
04	510
05	510
06	500
07	510
08	510
09	460

Table 4.6 Results of DTA – TG

Sample	II A slope	II B slope	II slope	Transition temperature (°c)
01	-0.032	0.100	0.101	168.63
02	0.045	0.110	0.120	175.03
03	0.010	0.120	0.124	149.60
04	0.080	0.117	0.121	178.14
05	0.020	0.136	0.139	161.52
06	-0.010	0.114	0.137	118.69
07	0.033	0.126	0.127	137.70
08	0.056	0.126	0.128	177.34
09	-0.073	0.179	0.144	177.63

CHAPTER – 5

ANALYSIS

Different graphs have been plotted between the various calculated parameters and are analysed individually.

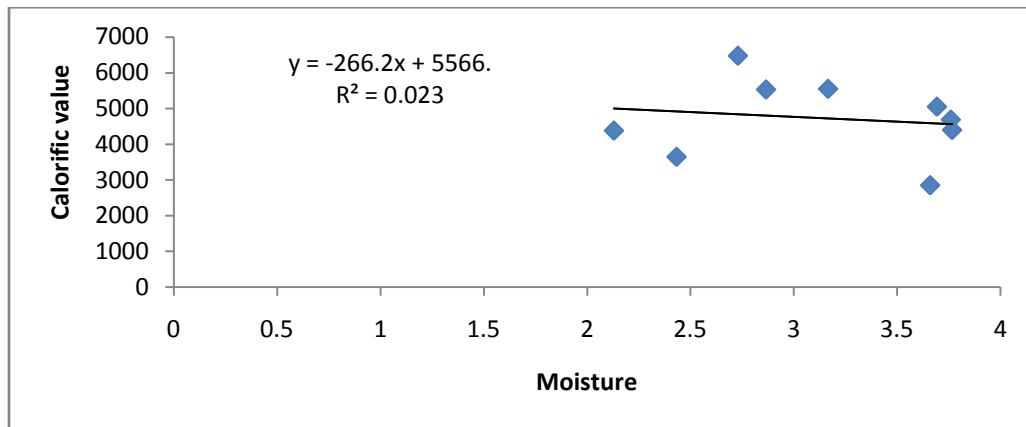


Fig: 5.1 Moisture Vs Calorific value

- Calorific value decreases with increase in moisture content.
- Fig. 5.1 shows that Calorific value has insignificant correlation with Moisture.

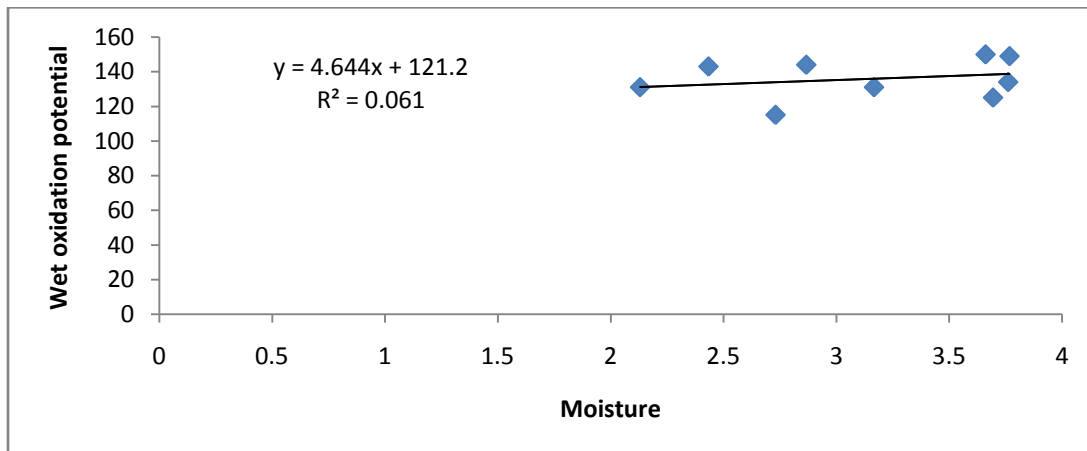


Fig: 5.2 Moisture Vs Wet oxidation potential

- Wet oxidation potential value slightly varies with increase in moisture.
- Wet oxidation potential value has insignificant correlation with moisture.

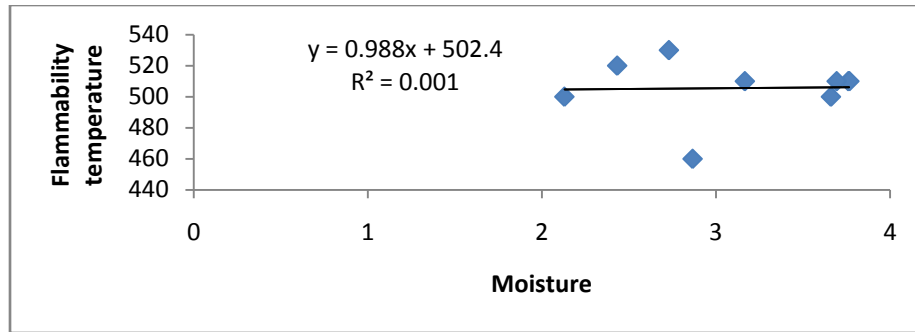


Fig: 5.3 Moisture Vs Flammability temperature

- Flammability temperature has insignificant correlation with moisture content
- Flammability temperature value increases with increase in moisture content

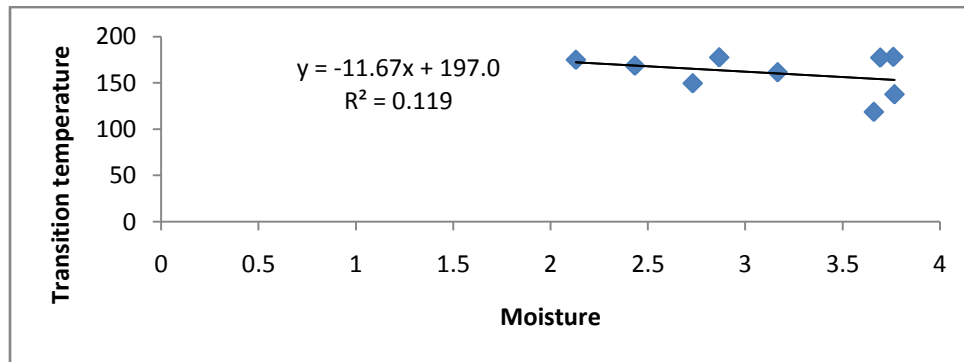


Fig: 5.4 Moisture Vs Transition temperature

- Transition temperature decreases with increase in moisture content.
- Transition temperature has significant correlation with moisture content.

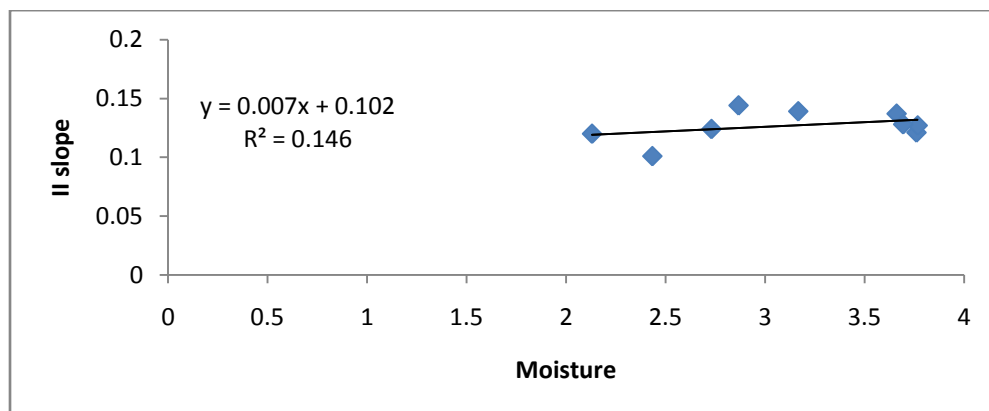


Fig: 5.5 Moisture Vs II slope of DTA

- As moisture content increases II slope of DTA also increases.
- Moisture content has insignificant correlation with II slope of DTA

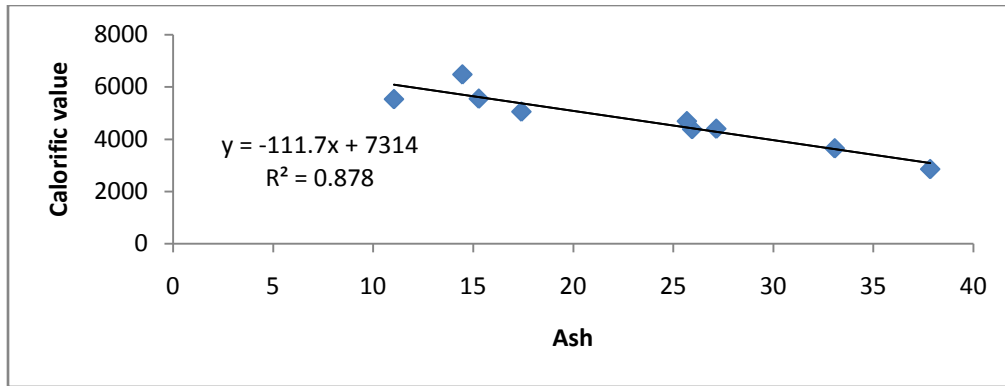


Fig: 5.6 Ash Vs Calorific value

- Ash content decreases with increase in calorific value.
- Ash content has insignificant correlation with calorific value.

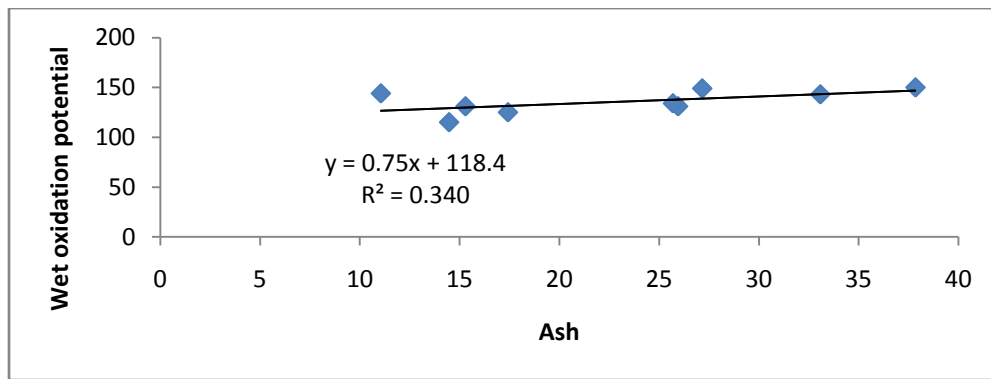


Fig: 5.7 Ash Vs Wet oxidation potential

- Ash content increases with increase in wet oxidation potential
- Ash content has insignificant correlation with wet oxidation potential

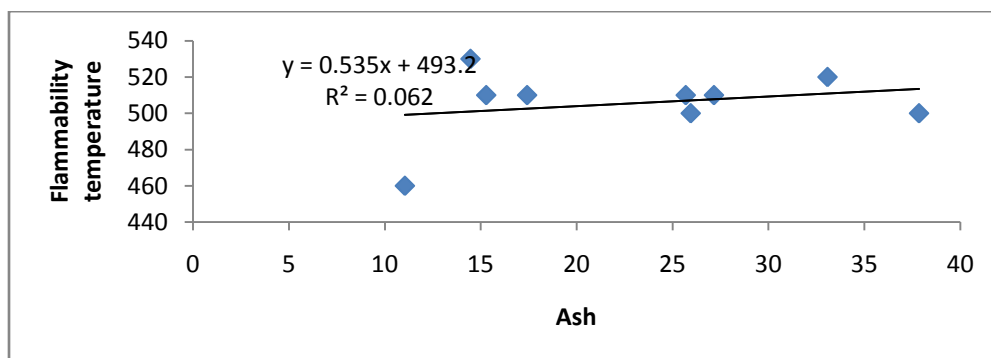


Fig: 5.8 Ash Vs Flammability temperature

- Ash content increases with increase in flammability temperature.
- Ash content has insignificant correlation with flammability temperature.

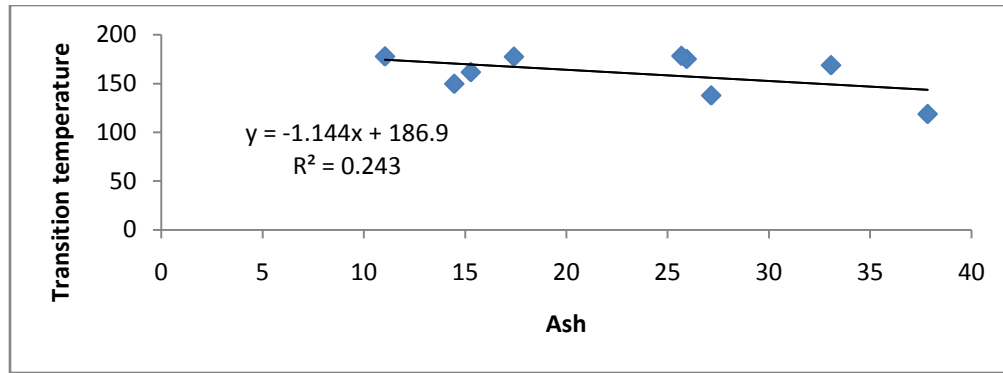


Fig: 5.9 Ash Vs Transition temperature

- Ash content is inversely proportional to transition temperature.
- Ash content has insignificant correlation with transition temperature.

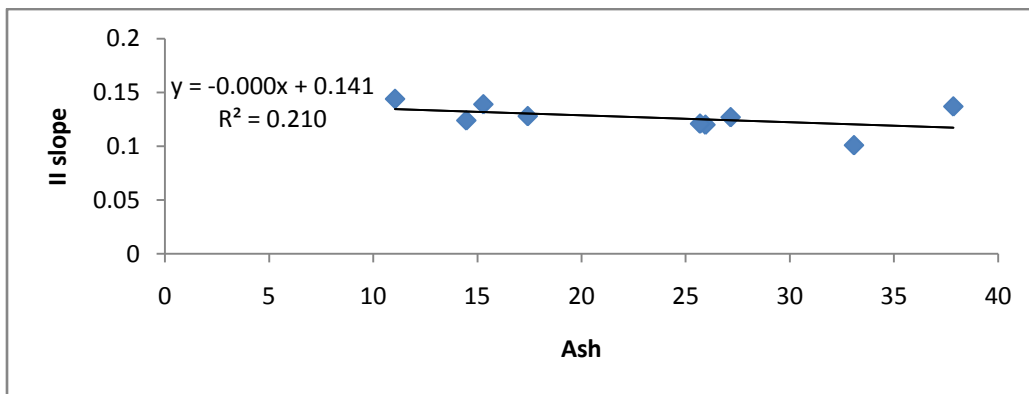


Fig: 5.10 Ash Vs II slope of DTA

- Ash content decreases with increase in the value of II slope of DTA.
- Ash content has insignificant correlation with II slope of DTA.

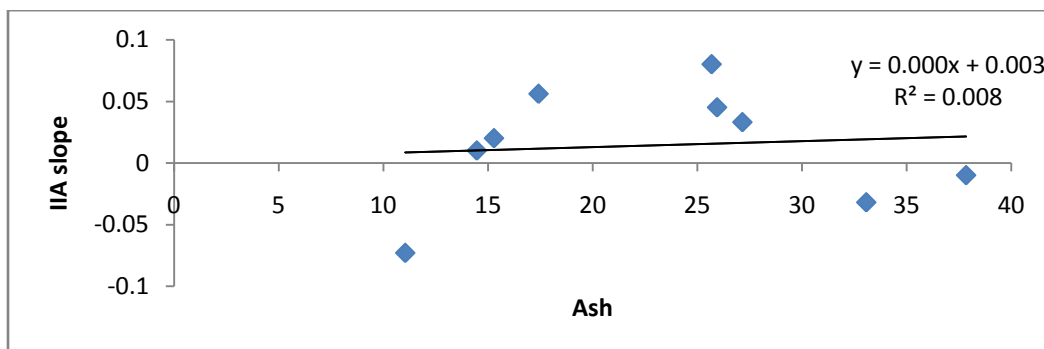


Fig: 5.11 Ash Vs IIA slope of DTA

- II A slope of DTA has insignificant correlation with ash content.
- As Ash content increases II A slope of DTA also increases.

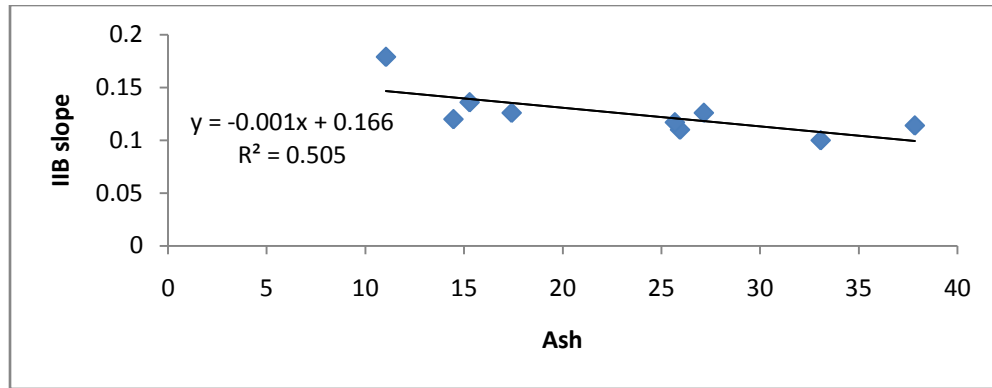


Fig: 5.12 Ash Vs IIB slope of DTA

- Ash content has significant correlation with II B slope of DTA.
- Ash content value increases with decrease in II B slope of DTA value.

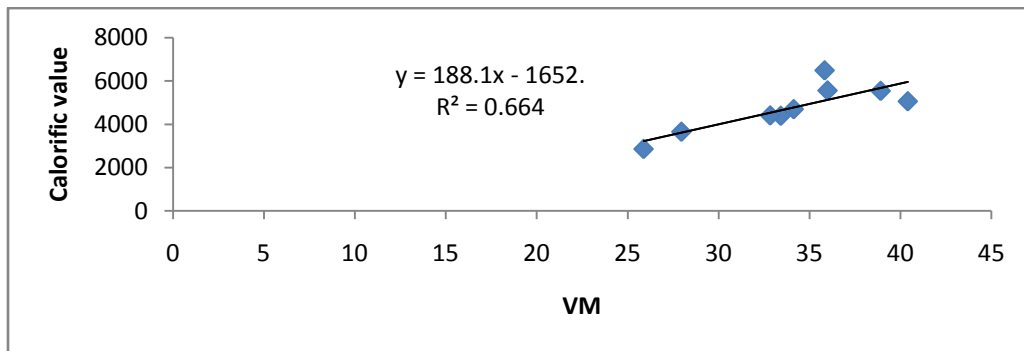


Fig: 5.13 VM Vs Calorific value

- Calorific value increases with increase in VM content.
- Calorific value has significant correlation with VM content.

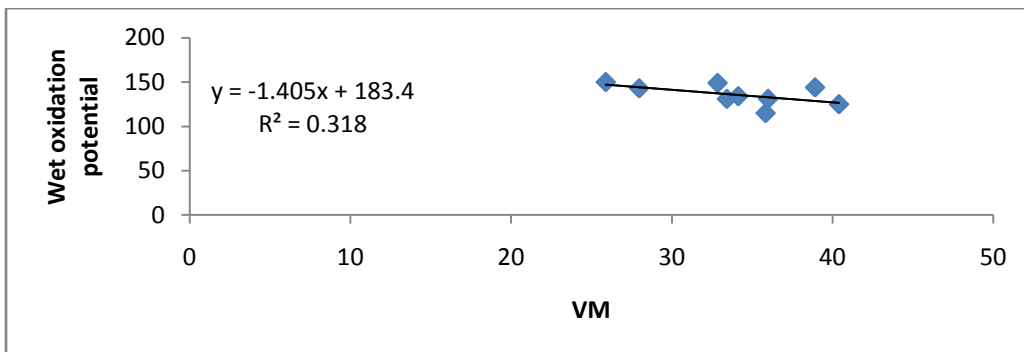


Fig: 5.14 VM Vs Wet oxidation potential

- Wet oxidation potential is inversely proportional to VM content.
- Wet oxidation potential has insignificant correlation with VM content.

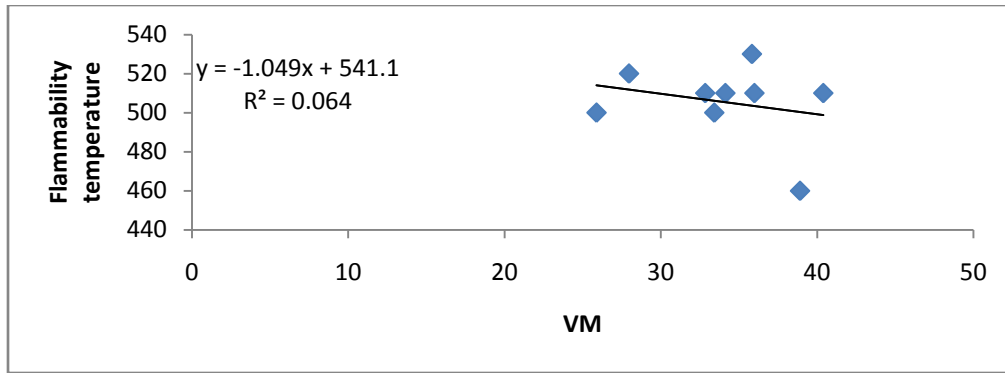


Fig: 5.15 VM Vs Flammability temperature

- Flammability temperature has insignificant correlation with VM content
- VM increases with decrease in the value of flammability temperature.

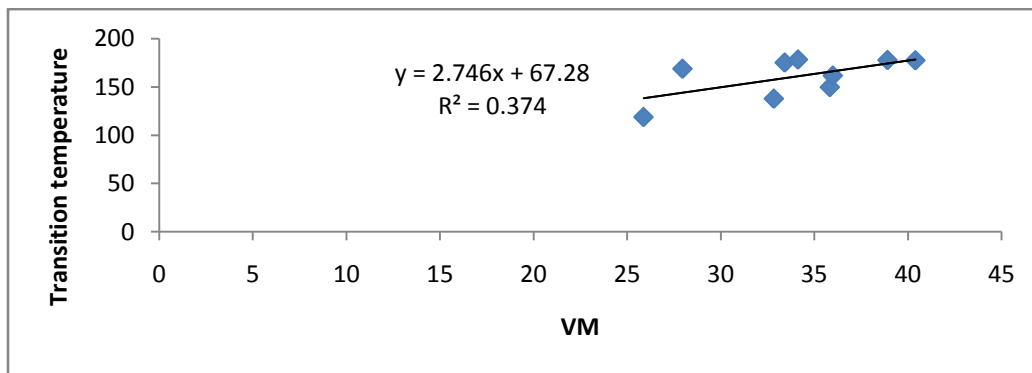


Fig: 5.16 VM Vs Transition temperature

- As VM content increases transition temperature increases so spontaneity decreases
- And has significant correlation than flammability temperature.

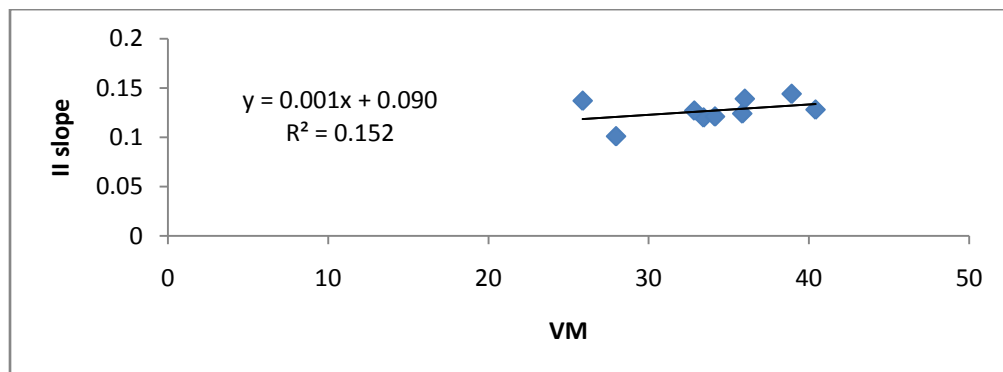


Fig: 5.17 VM Vs II slope of DTA

- VM content is directly proportional to II slope of DTA
- VM content increases with increase in the value of II slope of DTA.

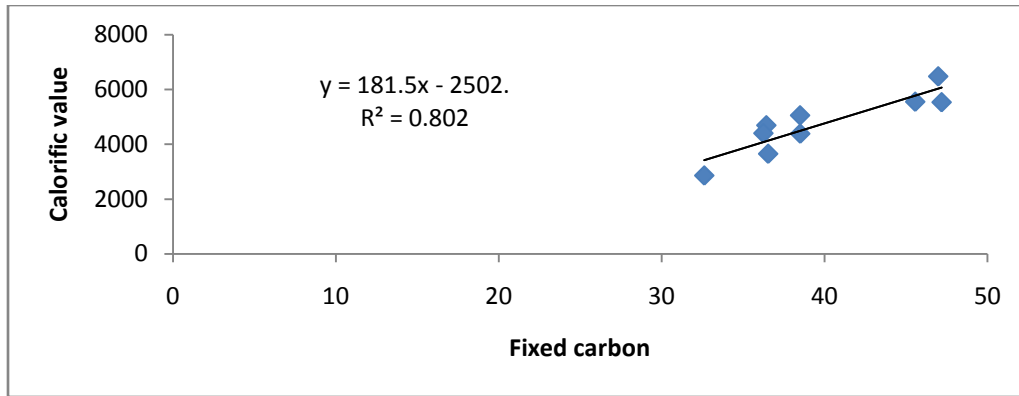


Fig: 5.18 Fixed carbon Vs Calorific value

- Calorific value increases with increase in fixed carbon value.
- Calorific value has significant correlation with fixed carbon value.

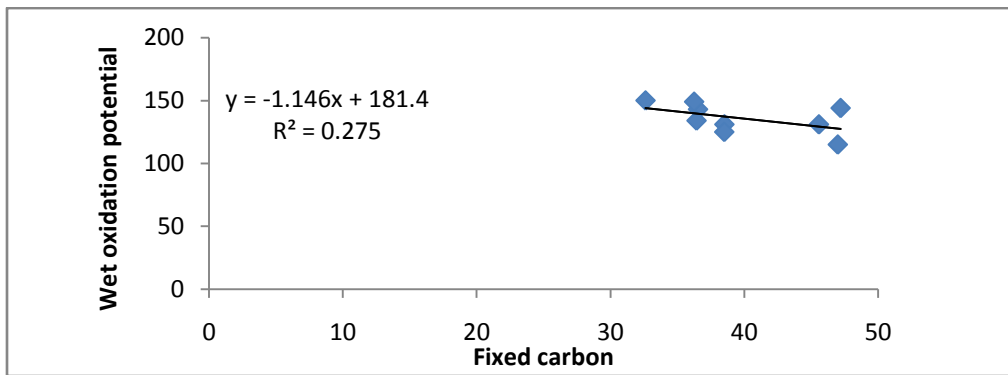


Fig: 3.19 Fixed carbon vs Wet oxidation potential

- Wet oxidation potential content decreases with increase fixed carbon content.
- Wet oxidation potential content has insignificant correlation with fixed carbon content.

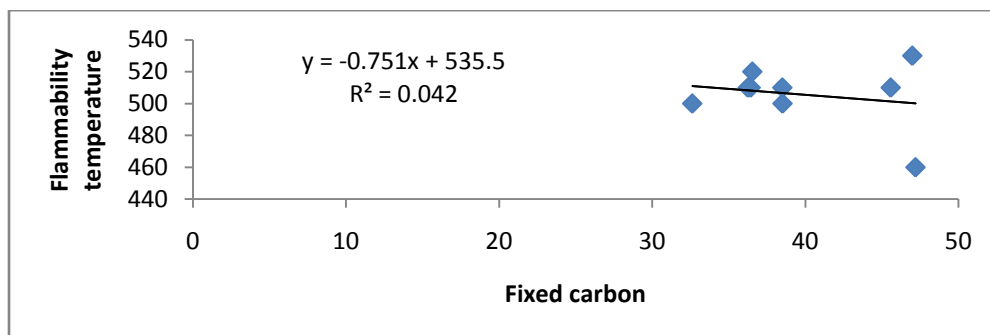


Fig: 5.20 Fixed carbon Vs Flammability temperature

- Flammability temperature has insignificant correlation with fixed carbon.
- Flammability temperature decreases with increase in fixed carbon content.

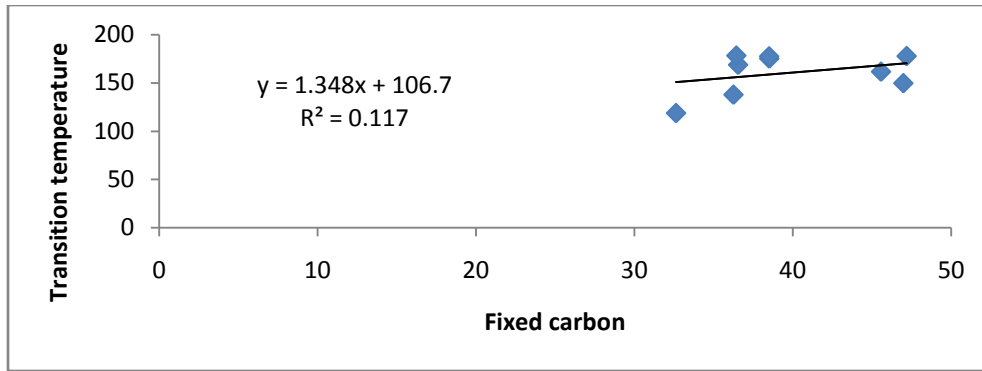


Fig: 5.21 Fixed carbon Vs Transition temperature

- Transition temperature increases with increase in fixed carbon content.
- Transition temperature has insignificant correlation with fixed carbon

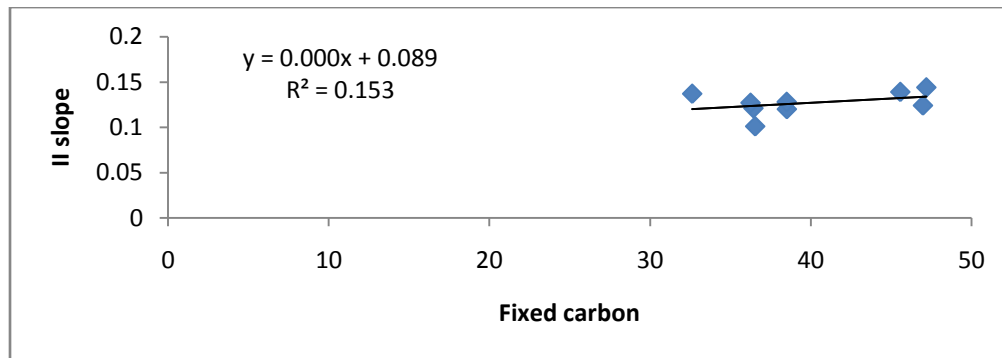


Fig: 5.22 Fixed carbon Vs II slope of DTA

- Fixed carbon content has insignificant correlation with II slope of DTA.
- Fixed carbon content increases as II slope of DTA increases.

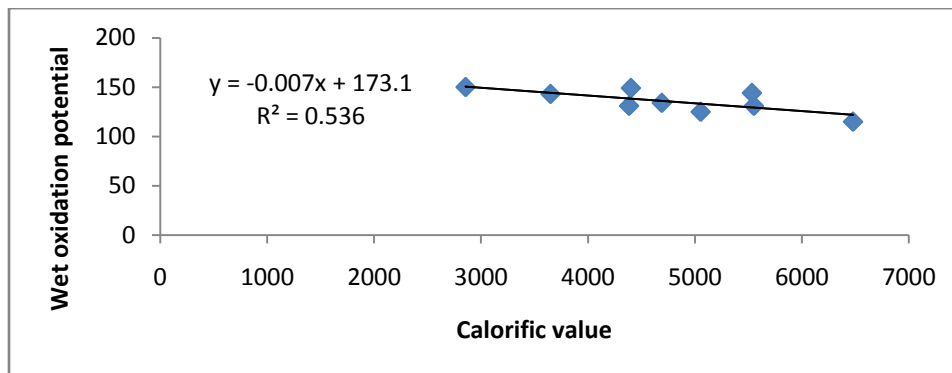


Fig: 5.23 Calorific value Vs Wet oxidation potential

- Wet oxidation potential has significant correlation with calorific value.
- Calorific value increases wet oxidation potential decreases.

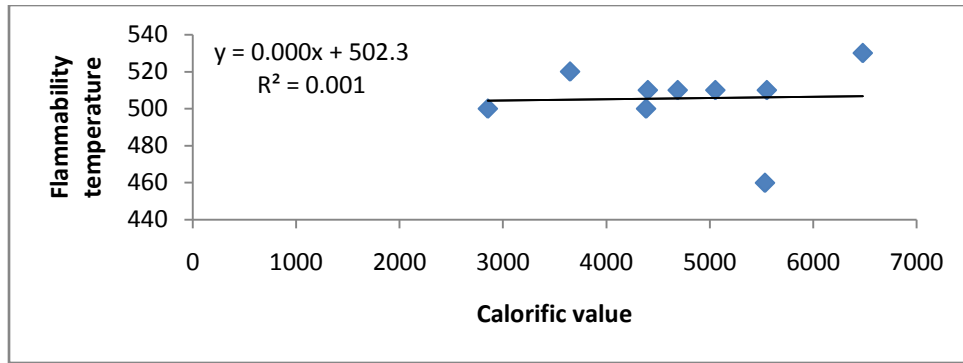


Fig: 5.24 Calorific value Vs Flammability temperature

- Calorific value has insignificant correlation with flammability temperature.
- Calorific value increases with increase in flammability temperature.

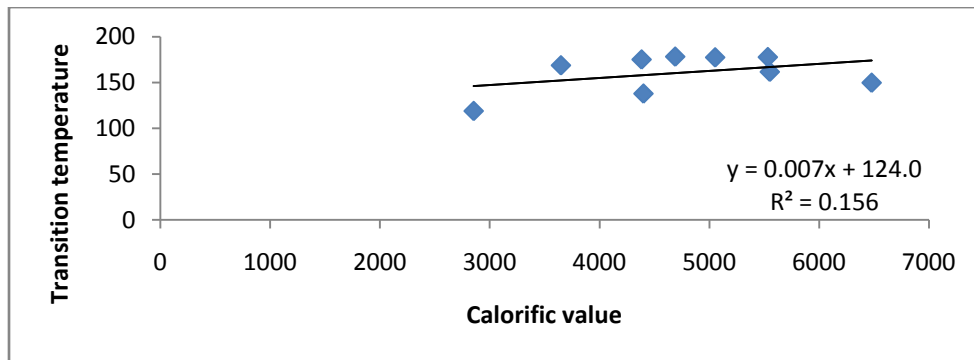


Fig: 5.25 Calorific value Vs Transition temperature

- Calorific value has insignificant correlation with Transition temperature.
- Calorific value increases with increase in Transition temperature.

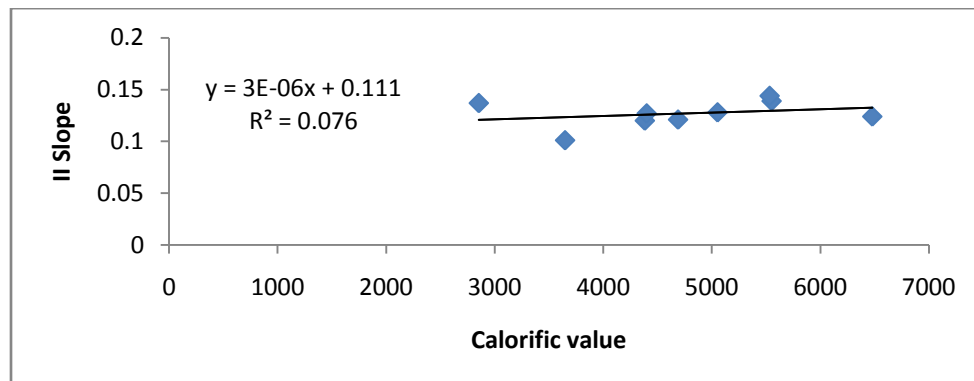


Fig: 5.26 Calorific value Vs II slope of DTA

- Calorific value increases with increase in II slope of DTA
- Calorific value has insignificant correlation with II slope of DTA.

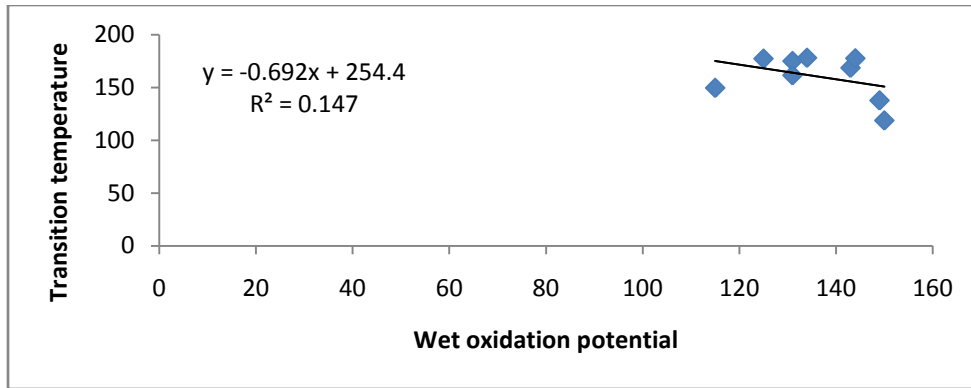


Fig: 5.27 Wet oxidation potential Vs Transition temperature

- Wet oxidation potential has insignificant correlation with Transition temperature.
- Transition temperature decreases with increase in wet oxidation potential value

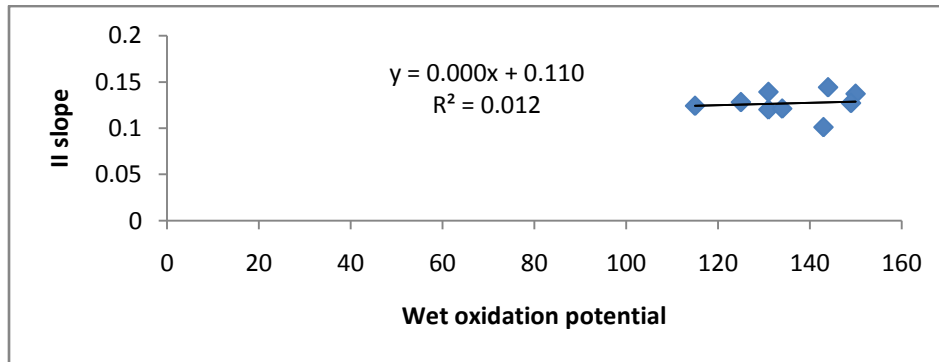


Fig: 5.28 Wet oxidation potential Vs II slope of DTA

- Wet oxidation potential increases with increase in II slope of DTA.
- Wet oxidation potential has insignificant correlation with II slope of DTA.

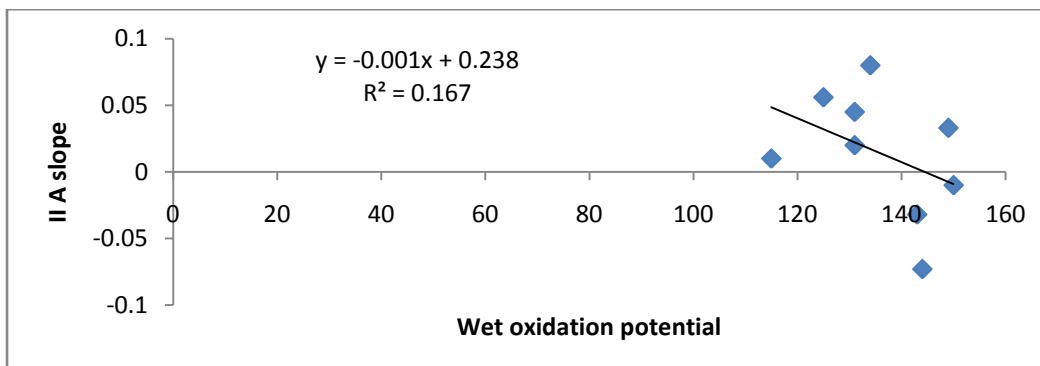


Fig: 5.29 Wet oxidation potential Vs IIA slope of DTA

- With increase in wet oxidation potential value II A slope value decreases.
- Wet oxidation potential has insignificant correlation with IIA slope of DTA.

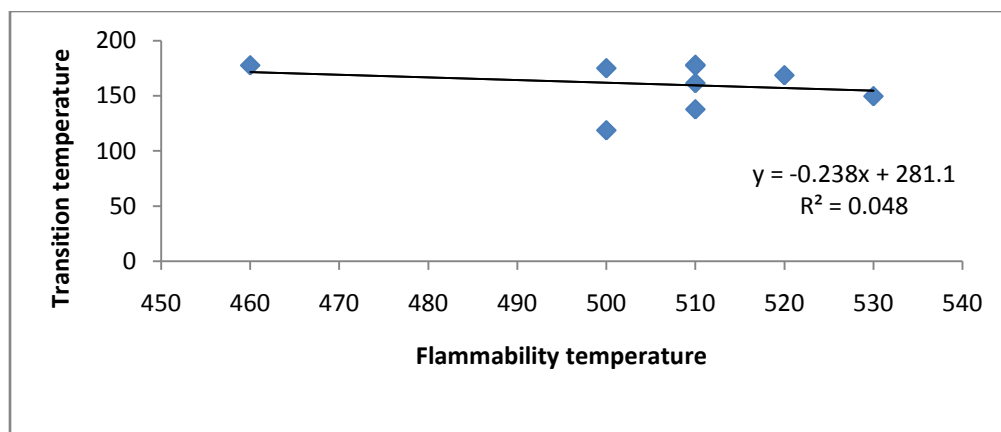


Fig: 5.30 Flammability temperature Vs Transition temperature

- Flammability temperature has insignificant correlation with transition temperature.
- Transition temperature value decreases with increase in flammability temperature.

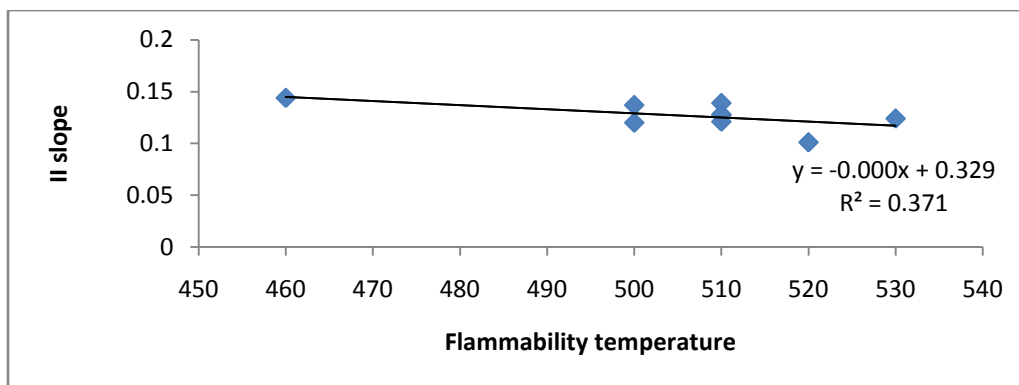


Fig: 5.31 Flammability temperature Vs II slope of DTA

- Flammability temperature increases with increase in II slope of DTA.
- Flammability temperature has insignificant correlation with II slope of DTA.

CHAPTER – 6

DISCUSSION AND CONCLUSIONS

In general, the following things are to be summarized from the results of all experiments. (Tables 4.2 to 4.6)

Discussion:

- The calorific value of sample 1 is found to be less so it has high susceptibility towards spontaneous heating.
- Sample 1 has “E” grade and its proximate analysis, EMF and Flammability temperature has moderate impact for spontaneity.
- Calorific value of sample 2 is found to be medium which shows that it has moderately susceptible to spontaneous heating.
- Sample 2 has “C” grade coal which shows poorly susceptible in nature.
- An experimental result shows that sample 3 is poorly susceptible and sample 4 is moderately susceptible to spontaneous heating.
- Sample 5 shows poor impact towards spontaneous combustion of coal.
- Sample 6 has low calorific value and low transition temperature which shows highly susceptible.
- An experimental result shows that sample 7 is moderately susceptible and sample 8 is poorly susceptible to spontaneous heating.
- Results of Flammability temperature and Calorific value of sample 9 shows that it is moderately susceptible to spontaneous heating.

Conclusions:

The following conclusions are drawn from the analysis of nine coal samples collected from SCCL:

1. Different graphs plotted between various parameters of experimental results show that

- The moisture content of coal decreases with increase in transition temperature.
- The calorific value of coal increases with increase in transition temperature.

2. Field observations and experimental results interpreted that the coal samples collected from the field are classified into the following category:

- Highly susceptible to spontaneous heating: Sample – 6.
- Moderately susceptible to spontaneous heating: Sample - 1, 2, 4 and 7.
- Poorly susceptible to spontaneous heating: Sample - 3, 5, 8 and 9

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APPENDIX

APPENDIX 1 - DTA CURVES

APPENDIX 2 - WET OXIDATION POTENTIAL CURVES

APPENDIX 1

DTA CURVES

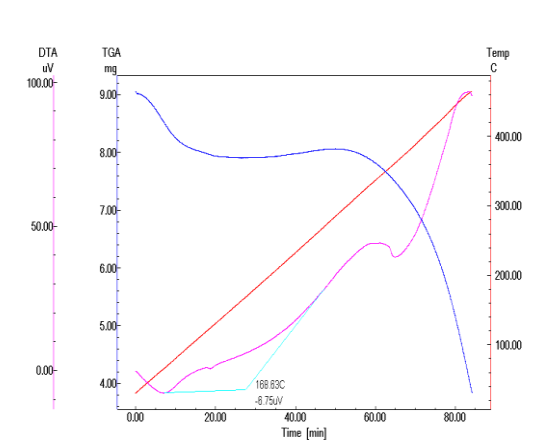


Fig A1: DTA-TG curve of sample 1

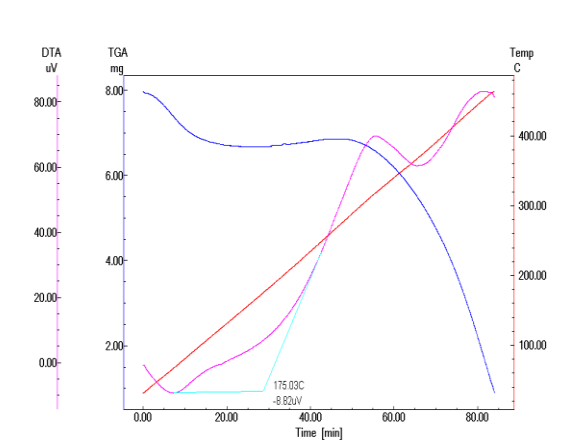


Fig A2: DTA-TG curve of sample 2

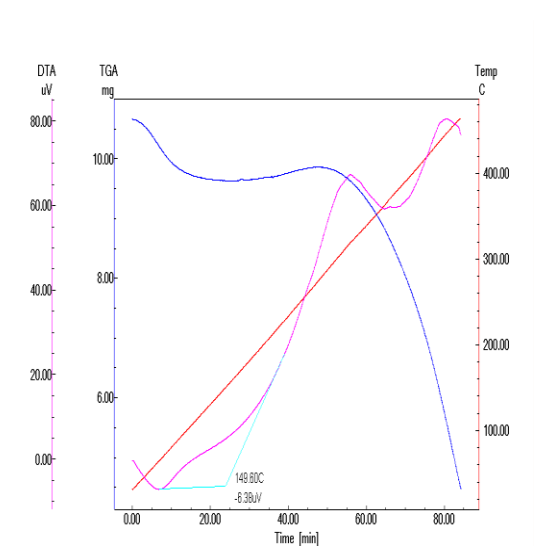


Fig A3: DTA-TG curve of sample 3

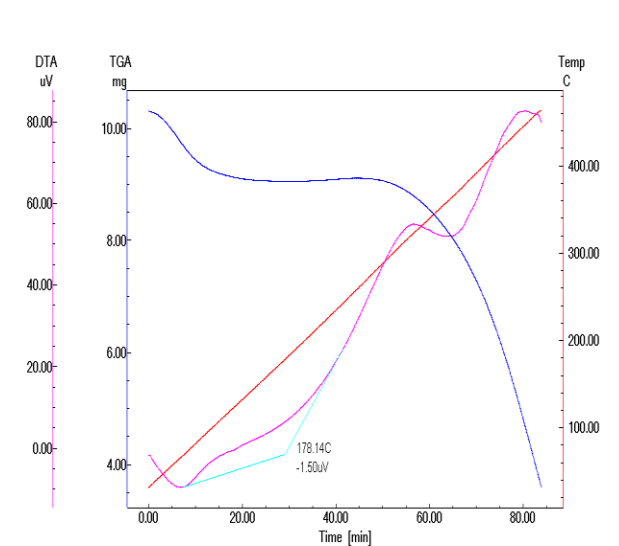


Fig A4: DTA-TG curve of sample 4

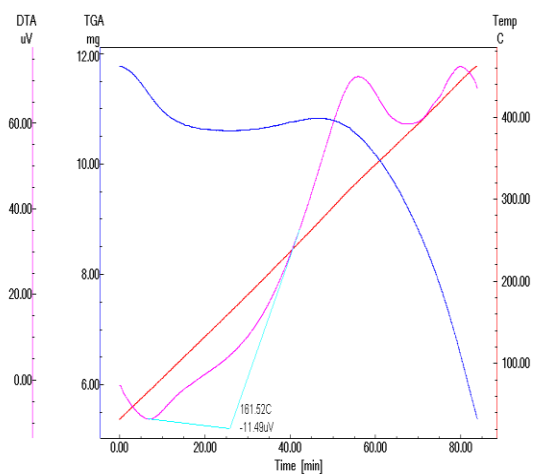


Fig A5: DTA-TG curve of sample 5

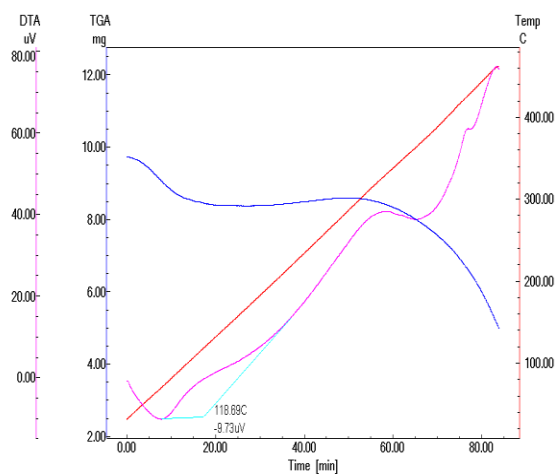


Fig A6: DTA-TG curve of sample 6

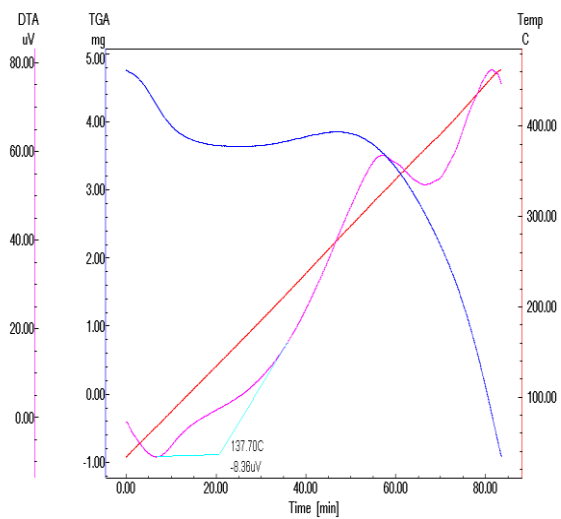


Fig A7: DTA-TG curve of sample 7

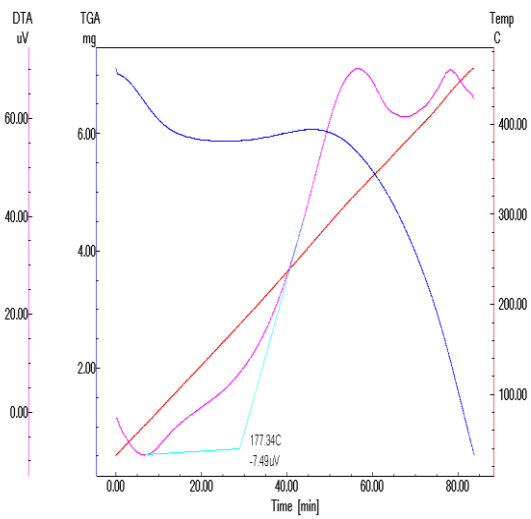


Fig A8: DTA-TG curve of sample 8

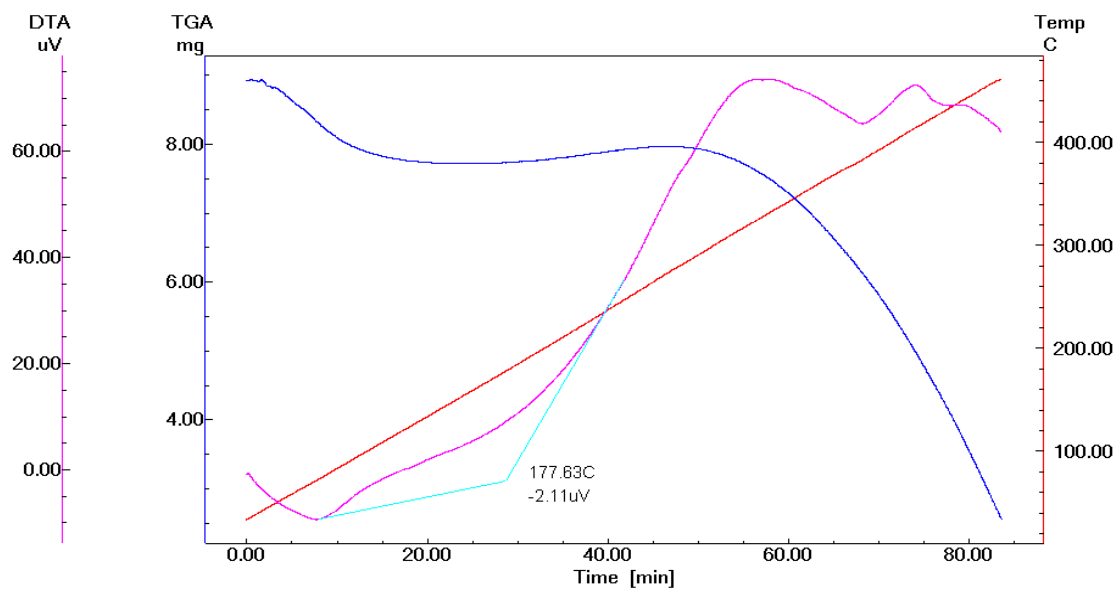


Fig A9: DTA-TG curve of sample 9

APPENDIX 2

WET OXIDATION POTENTIAL CURVES

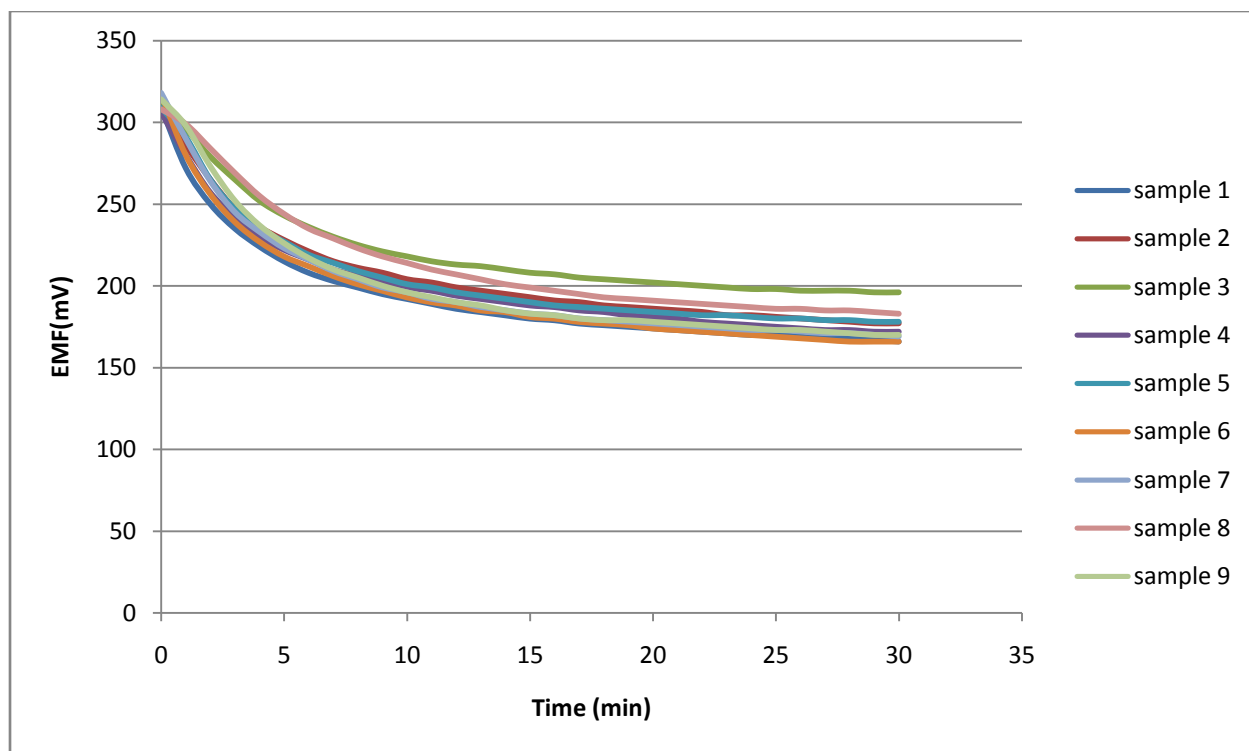


Fig A10: Wet oxidation potential curves